# A LIGA Fabricated Two-Dimensional Quadrupole Array for High Resolution Mass Spectroscopy

N. V. Myung, Otto Orient, Kirill Shcheglov,
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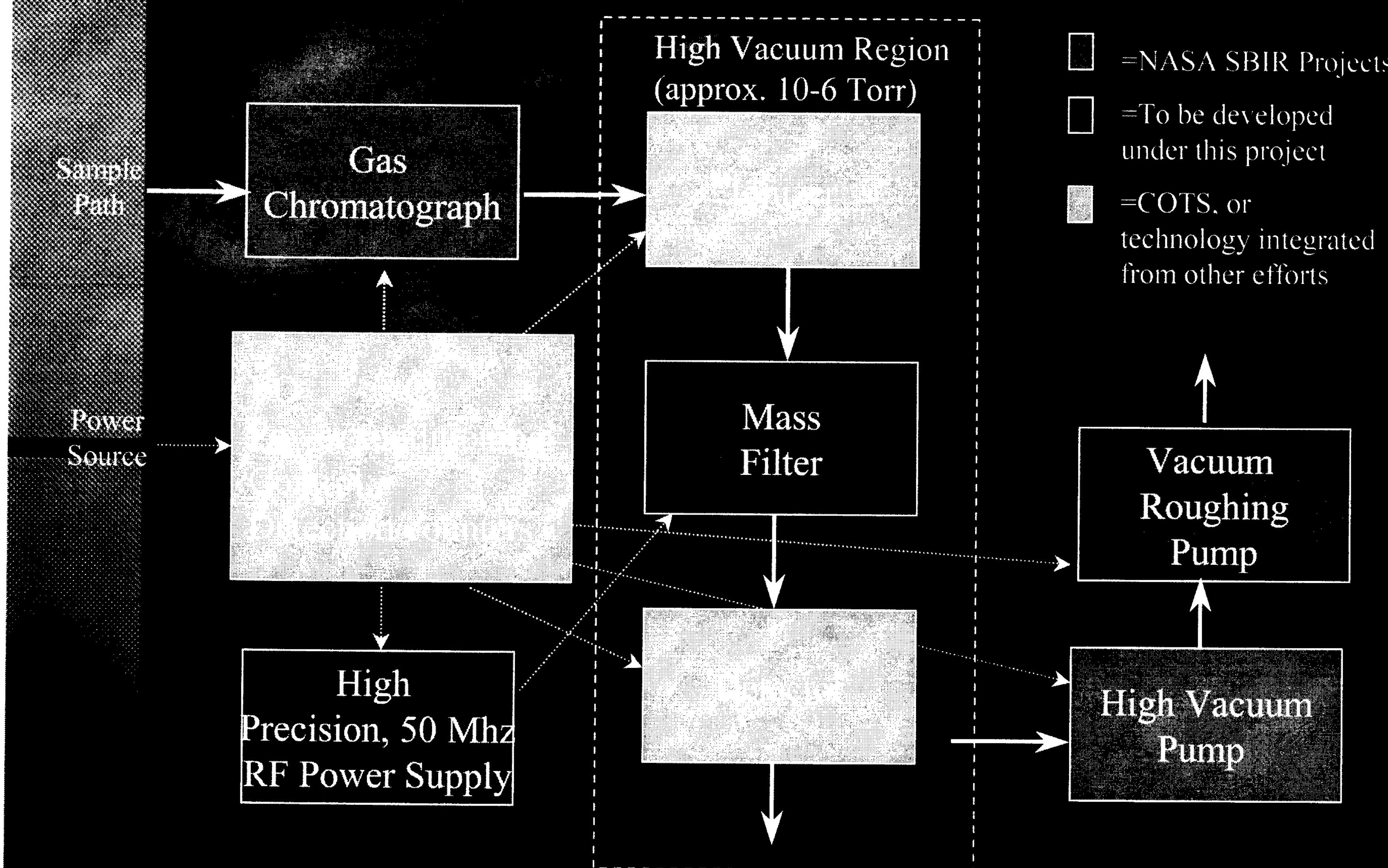
Jet Propulsion Laboratory,
California Institute of Technology

# Project Objectives and Approach:

The objective of this project is to build a functional miniaturized Quadrupole GC/MS system by advancing the state-of-the-art of key components which in turn will be integrated with other elements currently under development as NASA SBIR projects. This project seeks to leverage previous investments in NASA funded advanced technology projects to realize the functional components of the system.

# GC/MS System Block Diagram



# LIGA Processes

LIGA is a German acronym derived from **LI**thographie, **G**alvanoformung and **A**bformung which are interpreted as lithography, electroplating and replication. It is an X-ray lithography technique in which a polymer film is exposed to x-ray radiation which breaks some of the polymeric bonds causing a reduction in molecular weight of the exposed areas. This difference in molecular weight is then exploited to dissolve away the exposed areas leaving a mold into which metals are electroplated. This can be used as a final part or is the basis for further replication using techniques such as injection molding.

# Advantage of LIGA micromachining

- Able to produce complex microstructures
- High Precision(good dimension control)
  - Very small structures (10 micron range)
  - High Aspectio ratio (upto 100)
  - Vertical Sidewalls ( 1 micron / 1000 micron slope)
  - Very smooth sidewalls (variation less than 30 nm)

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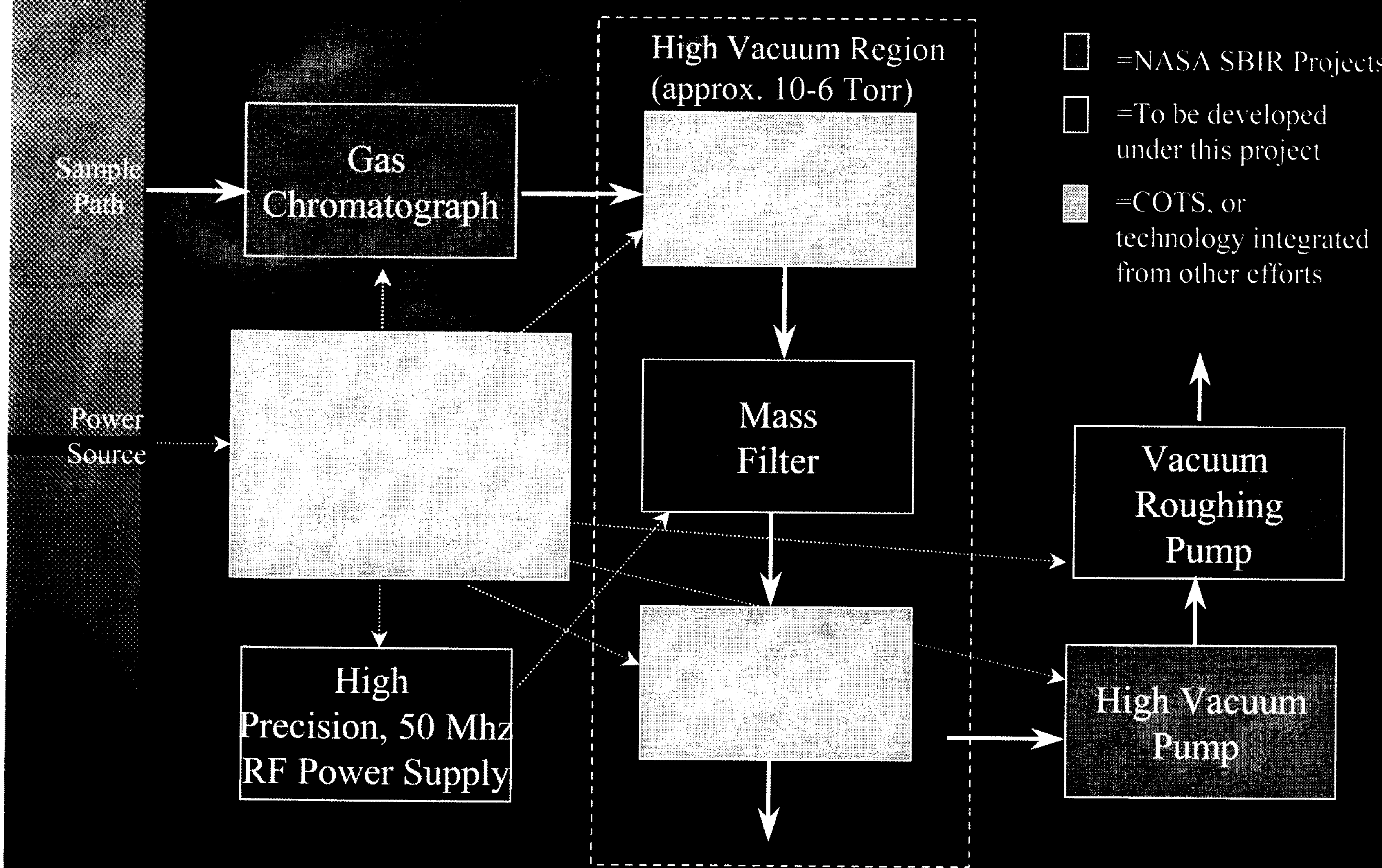

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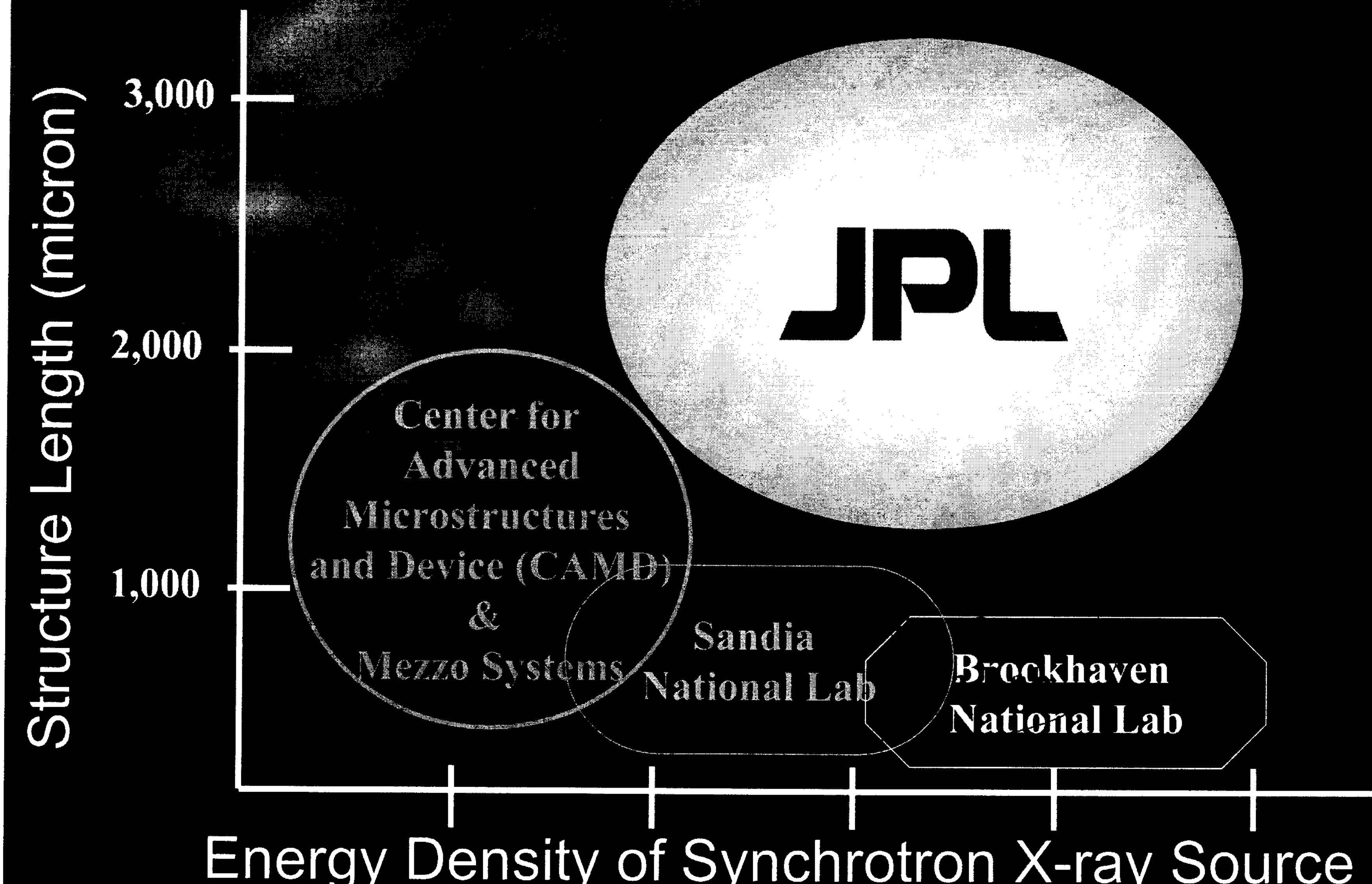

Structure Length (micron)
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2,000
1,000
JPL
Center for Advanced Microstructures and Device (CAMD) & Mezzo Systems
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Energy Density of Synchrotron X-ray Source

# Fabrication Steps

- Preparation of X-ray mask
  - Thick photoresist patterning on silicon wafer
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- PMMA bonding
- Synchrotron X-ray radiation of PMMA
  - Brake down of the long molecular chain (750,000 g/mol) to shorter chain (3000 to 6000 g/mol)
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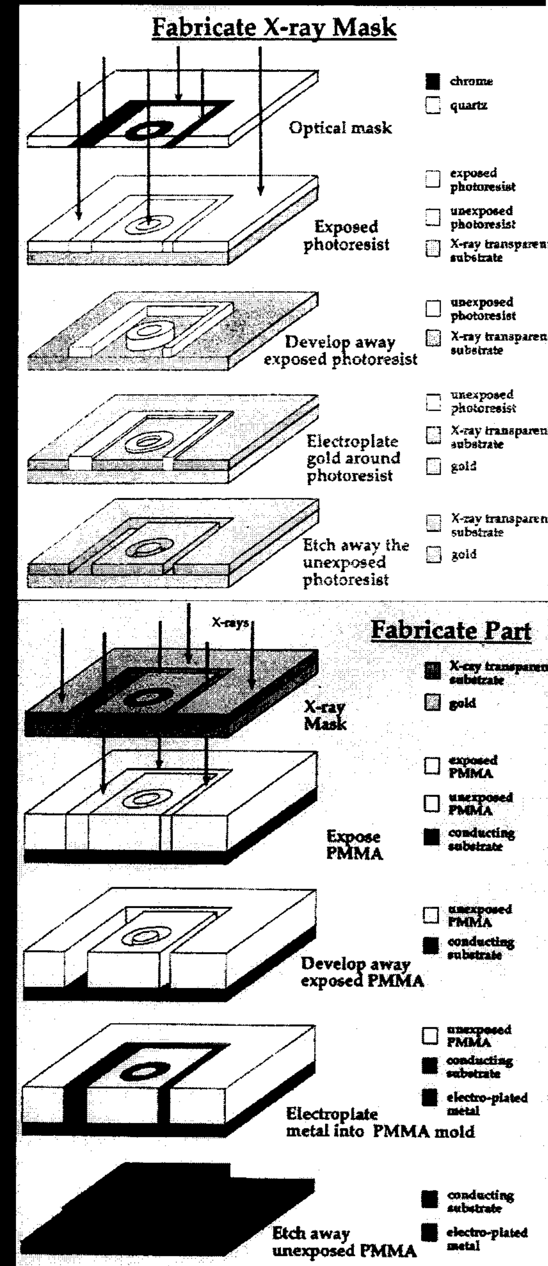

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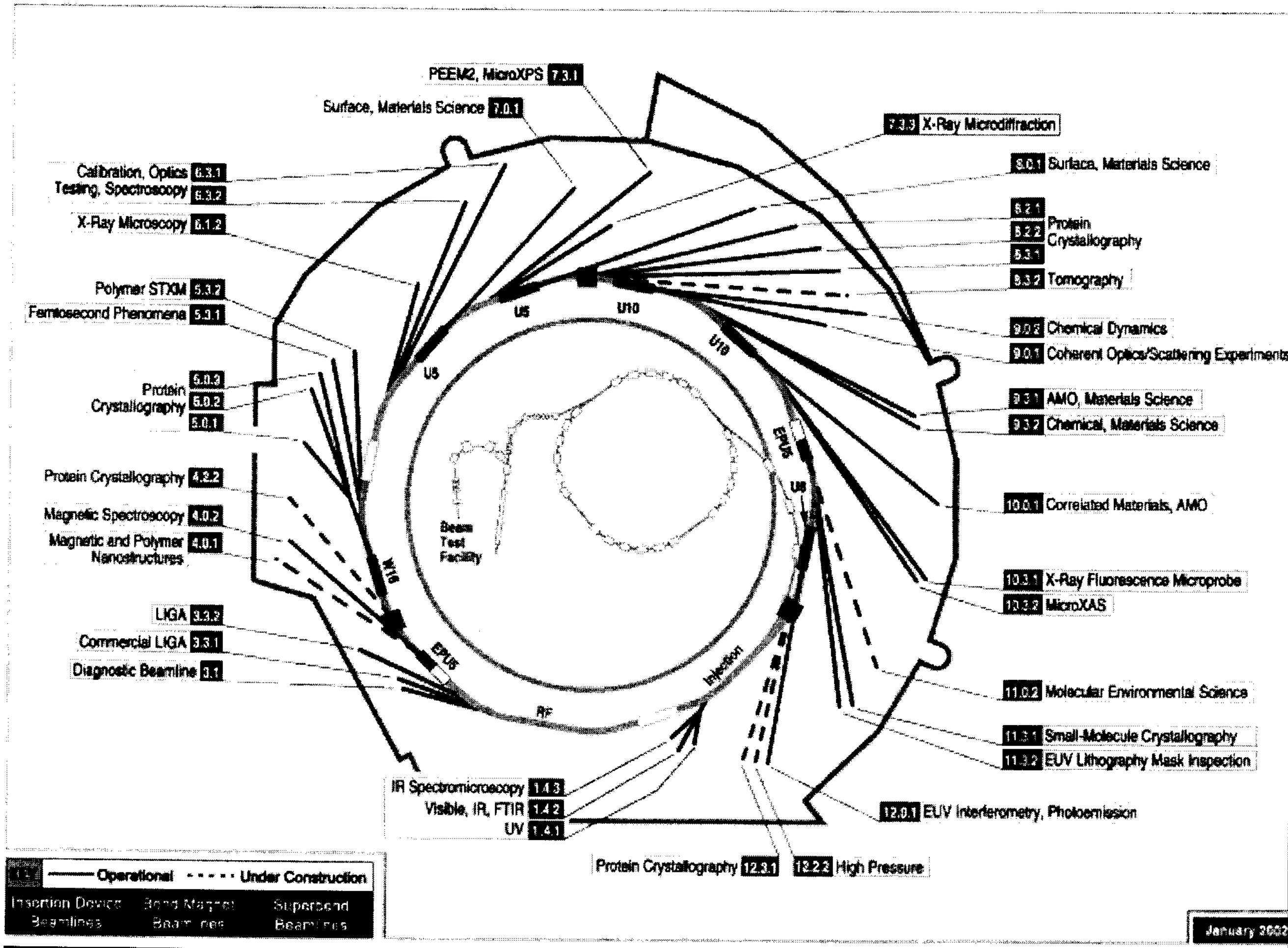
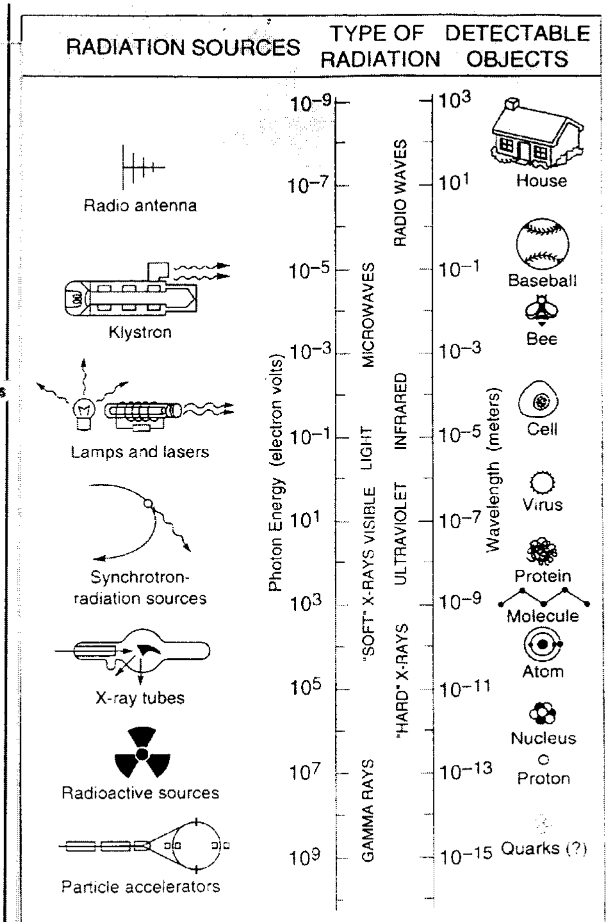
# Electroplated Gold Mask (X-ray absorber)

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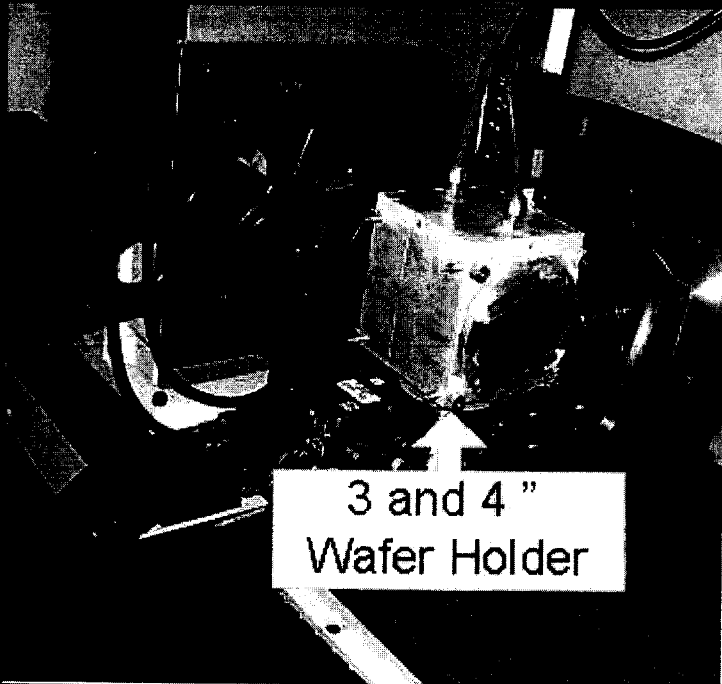
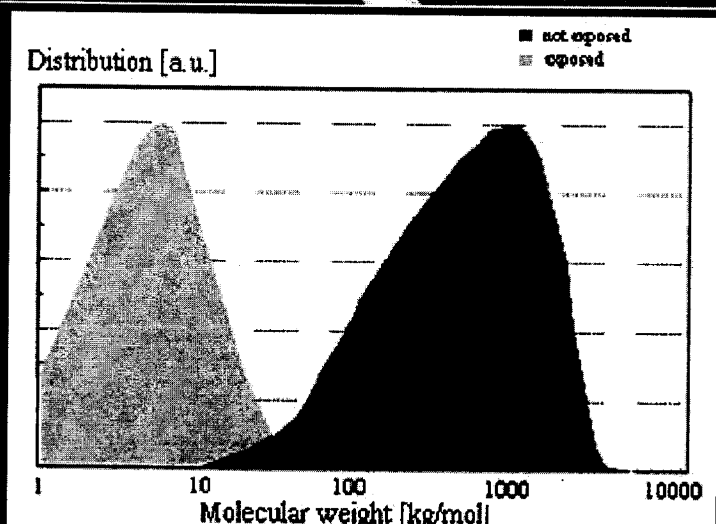
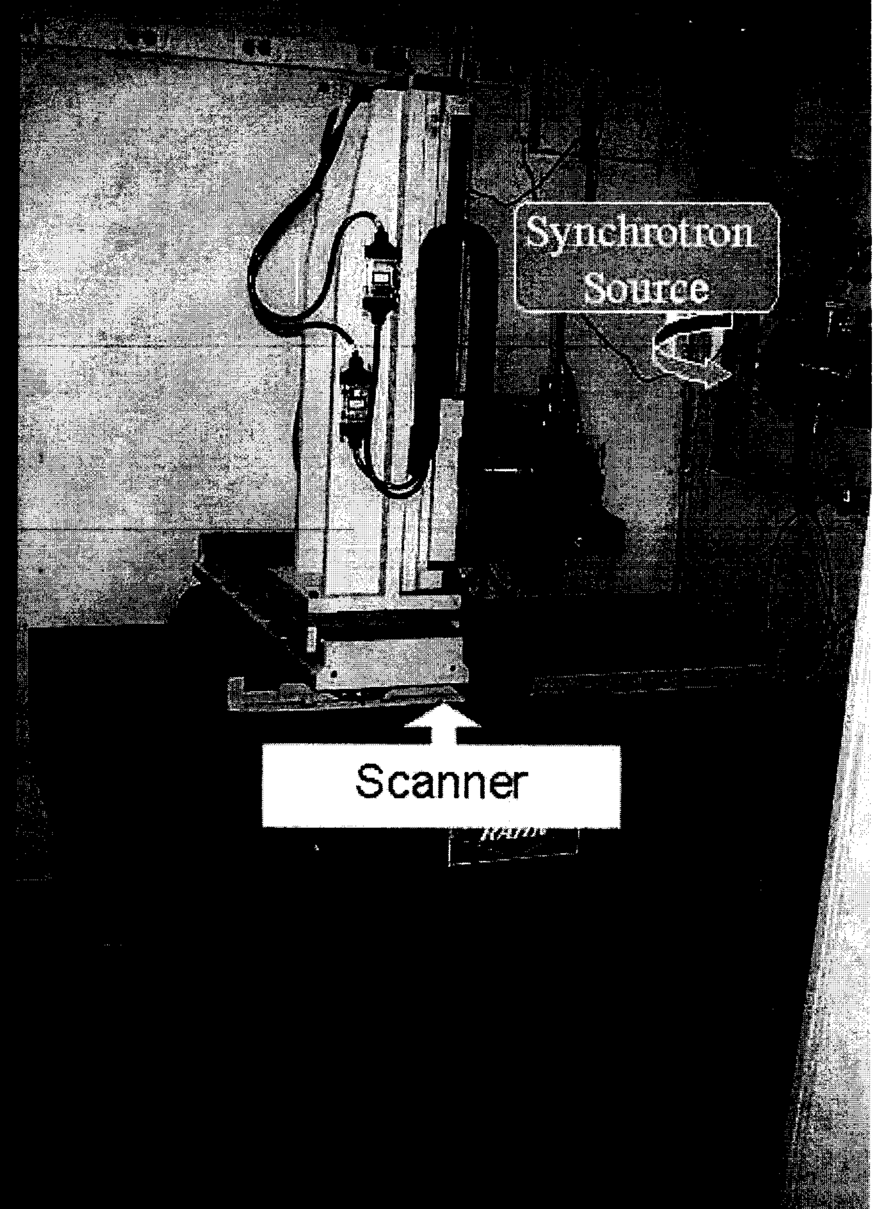
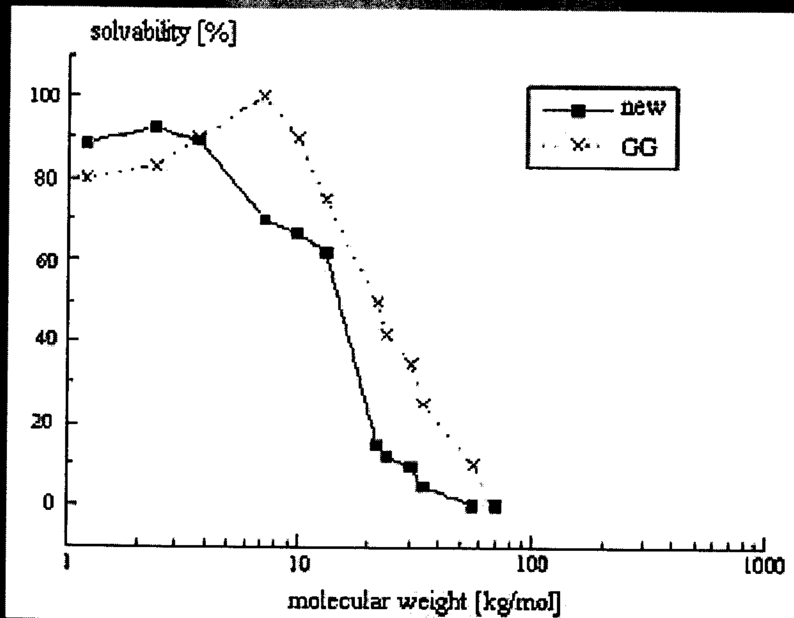
# Synchrotron X-ray exposure



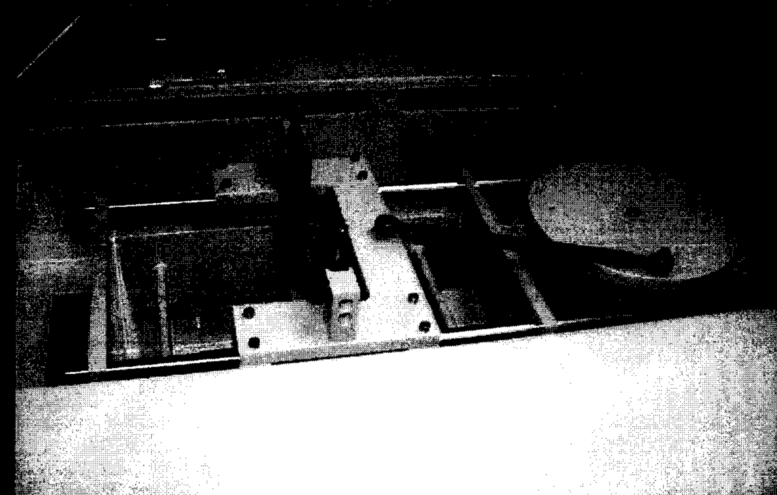



# Developing



solvability [%]
100
80
60
40
20
0
new
GG
1
10
100
1000
molecular weight [kg/mol]

# Electroplating

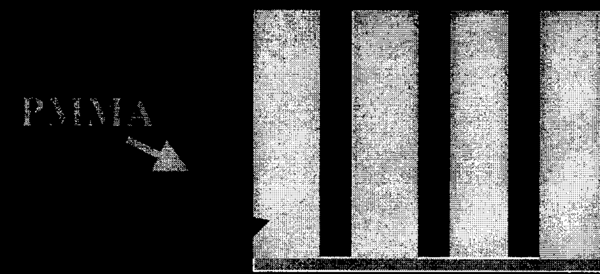


Electroplating Stations with fine temperature and process control

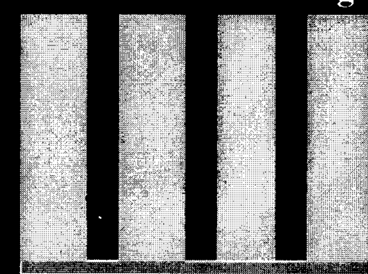
Electroplating Paddle Cell

# Electroplating

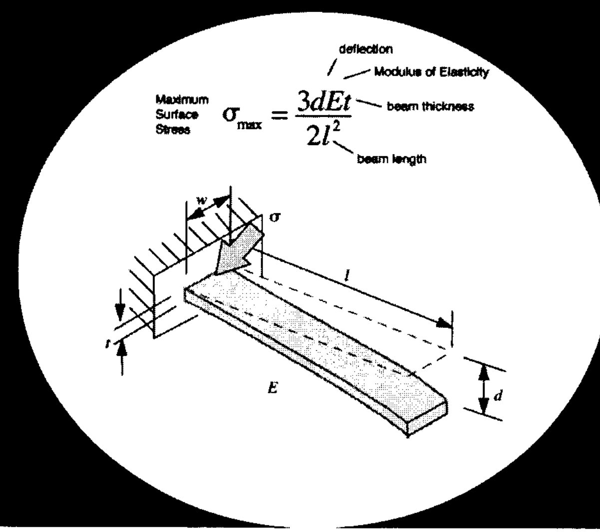
- Developing new electroplating solution to enhance mass transfer in deep trench and promote uniform growth (eliminate dendrite and powdery growth)
- Developing electroplating solution and processes to minimize internal stress in the deposits



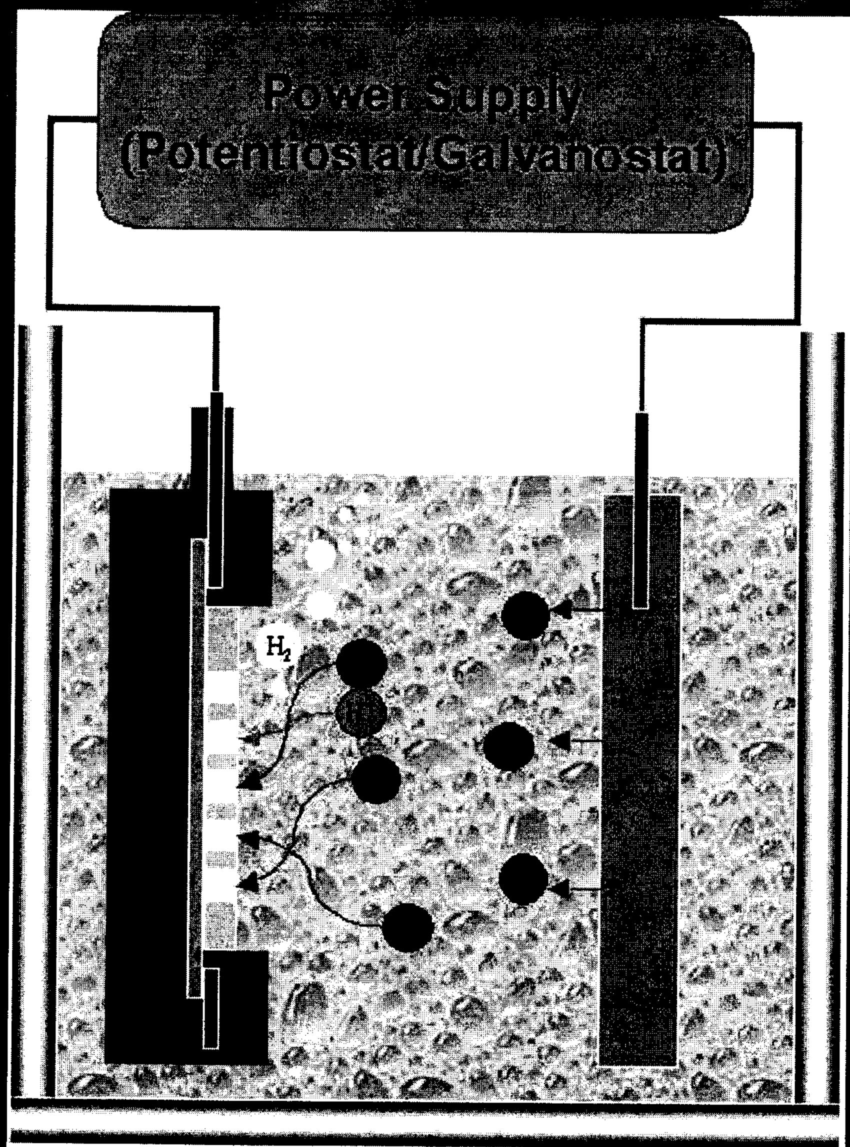

Uniform 2-dimensional growth



Non-uniform dendrite or powdery growth



# Electroplating apparatus




Cathodic(Reduction) Reaction

e.g.

$Ni^{+2} + 2e^- \rightarrow Ni_{(s)}$

$2H^+ + 2e^- \rightarrow H_2$ (side reaction)

Anodic(Oxidation) Reaction

e.g.

$Ni_{(s)} \rightarrow Ni^{+2} + 2e^-$

Anode

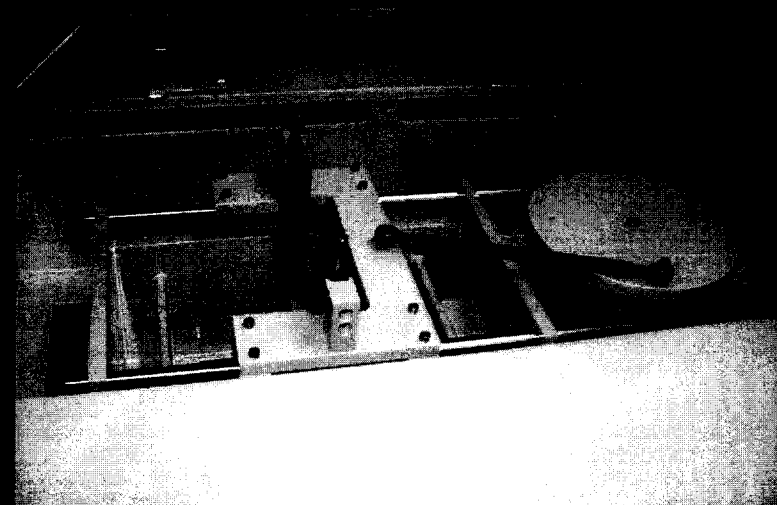
Substrate

PMMA (mold)

Substrate holder

# Electroplating

Electroplating Stations with fine temperature and process control



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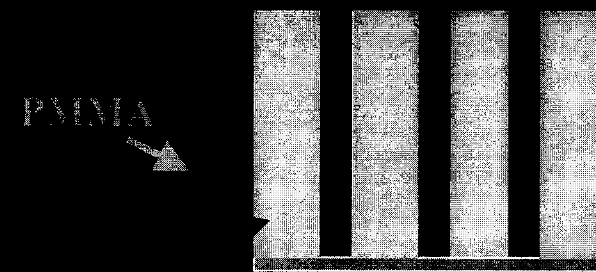
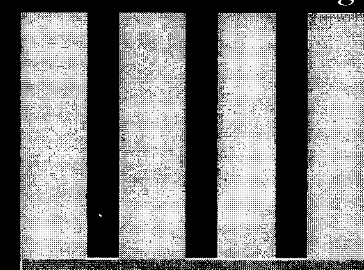
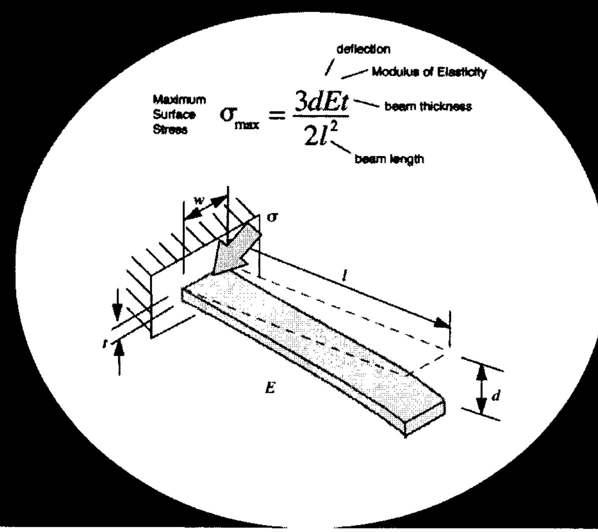


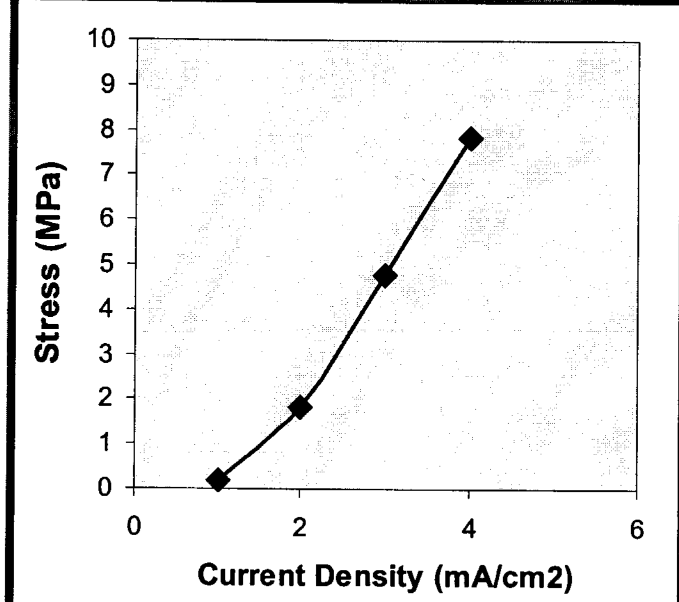

Uniform 2-dimensional growth

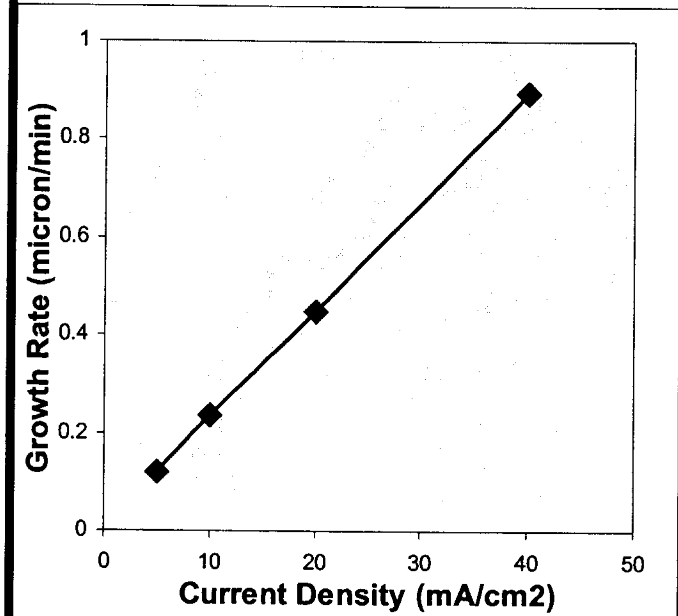


Non-uniform dendrite or powdery growth

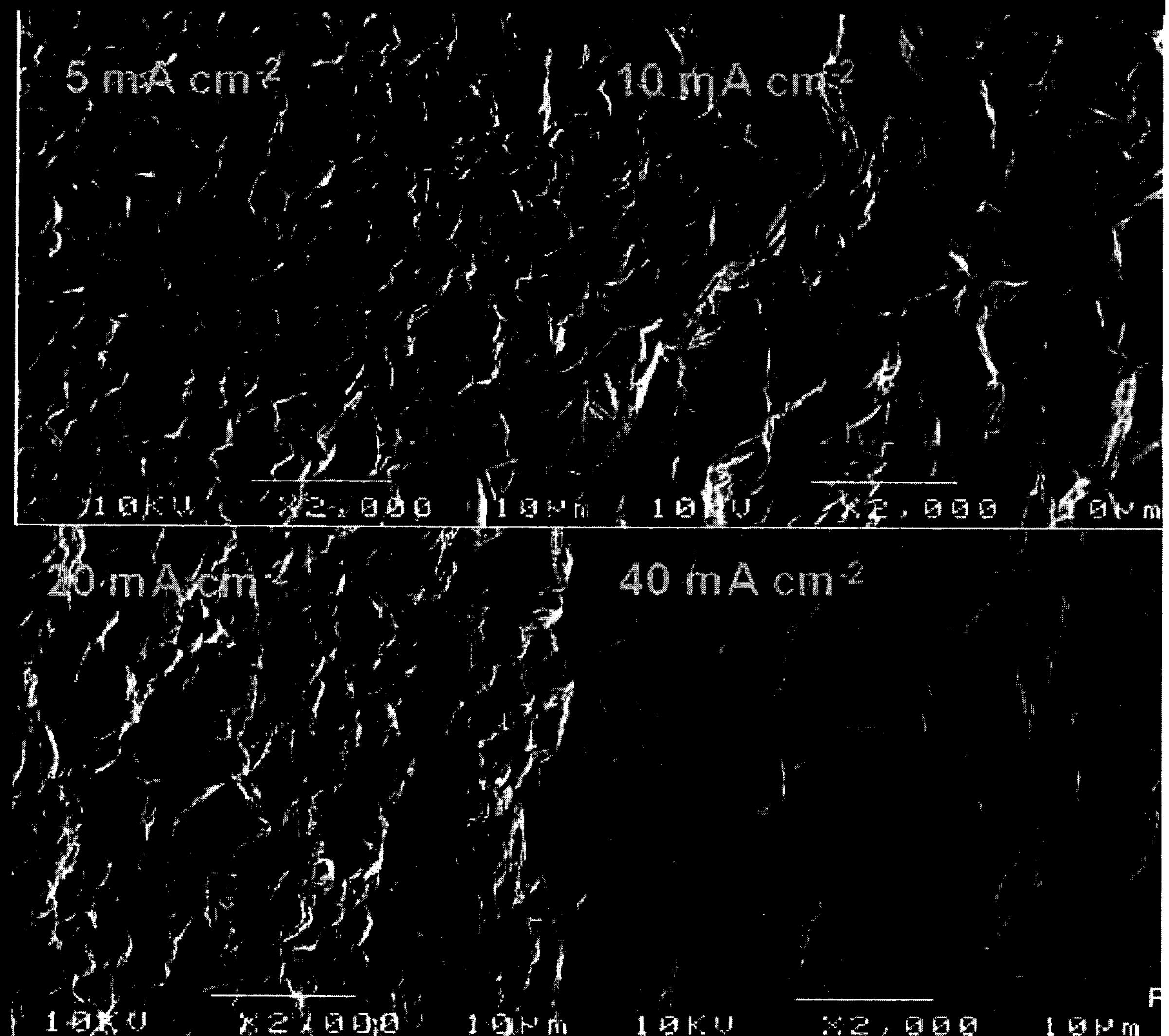


# Stress in Electroplated Cu




## SEM micrographs of electroplated Cu



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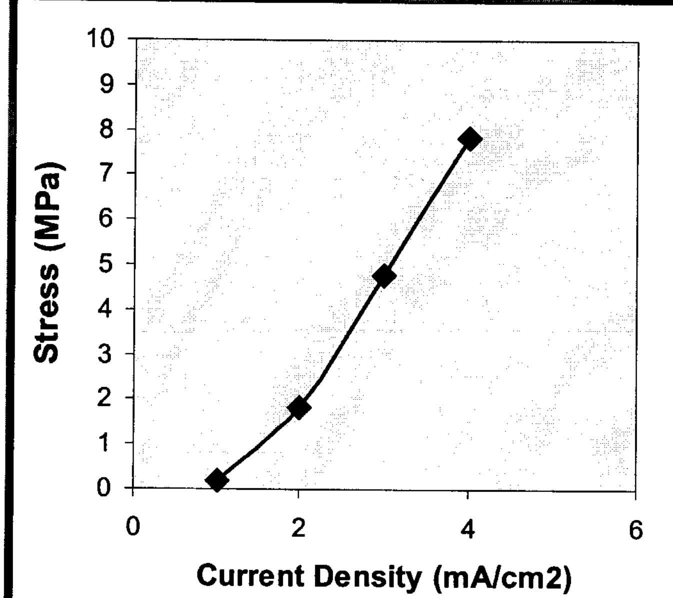
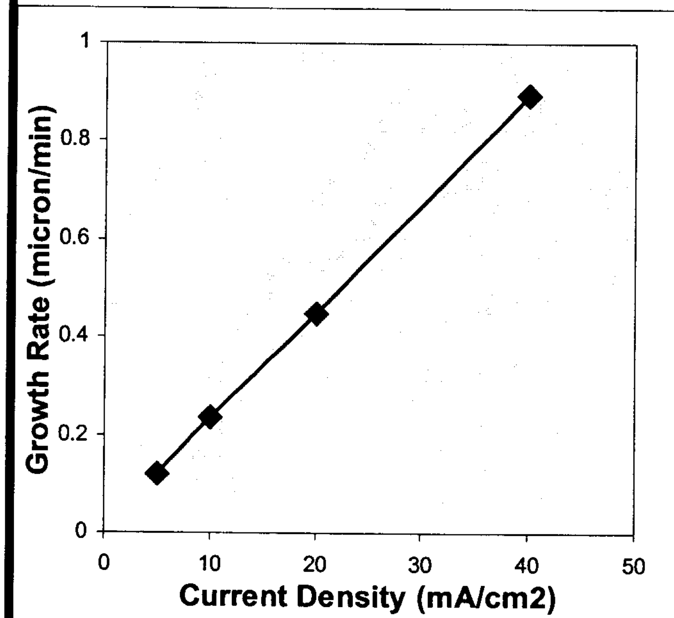
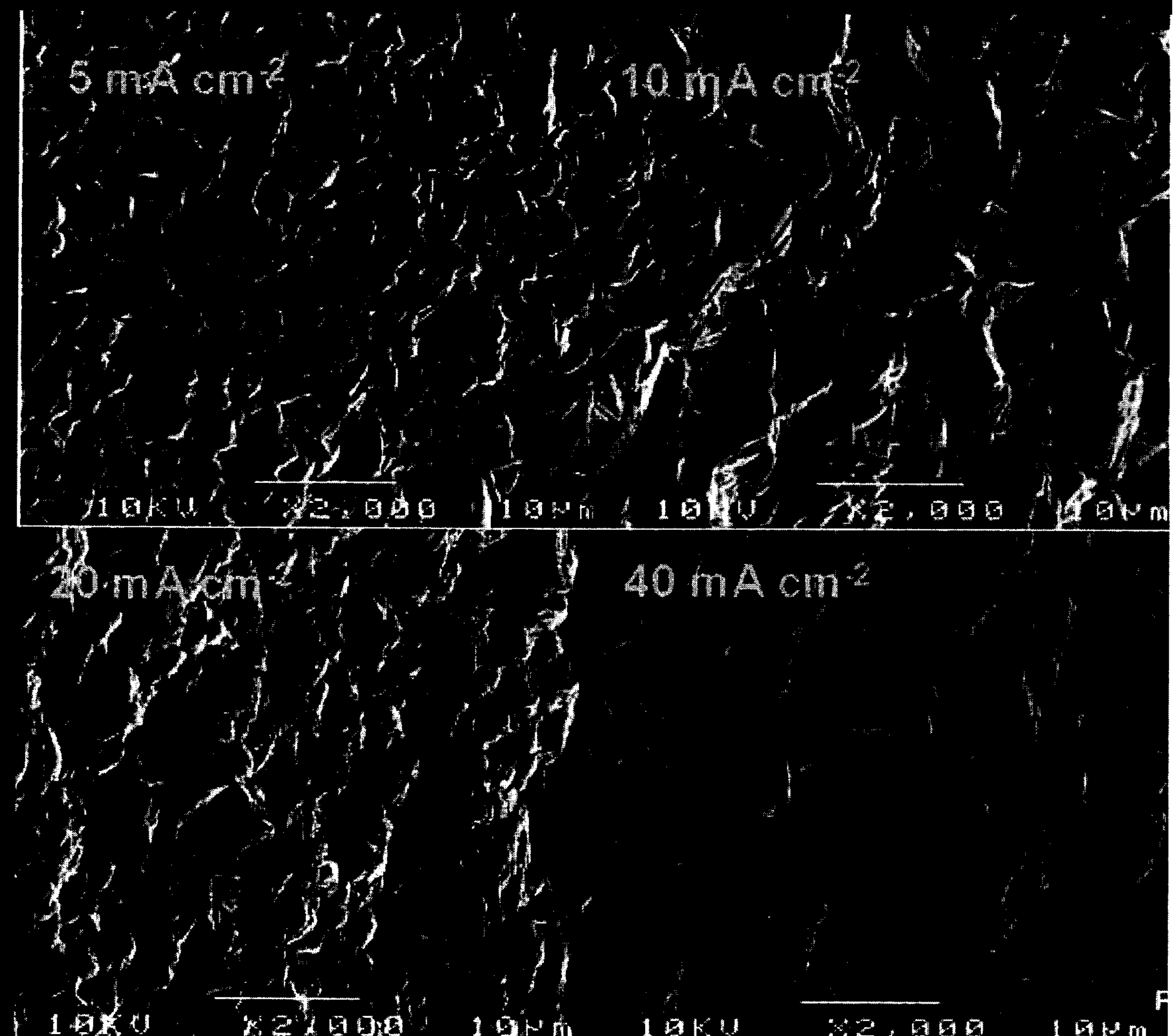
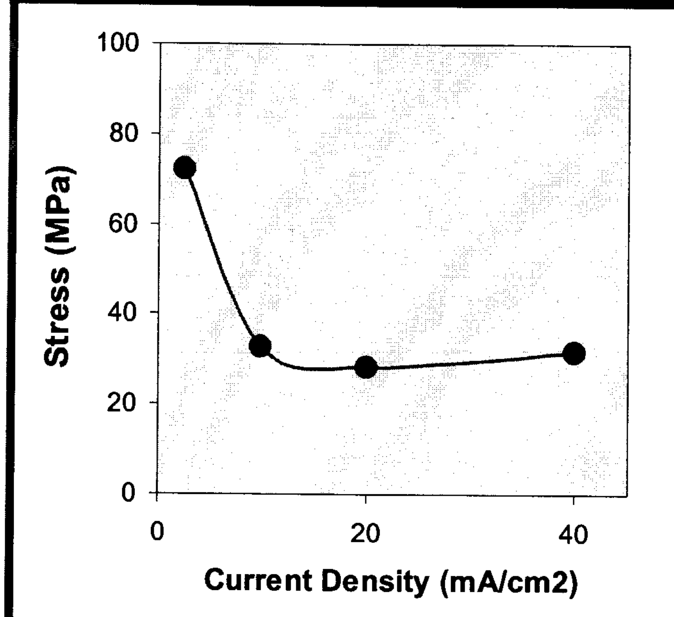


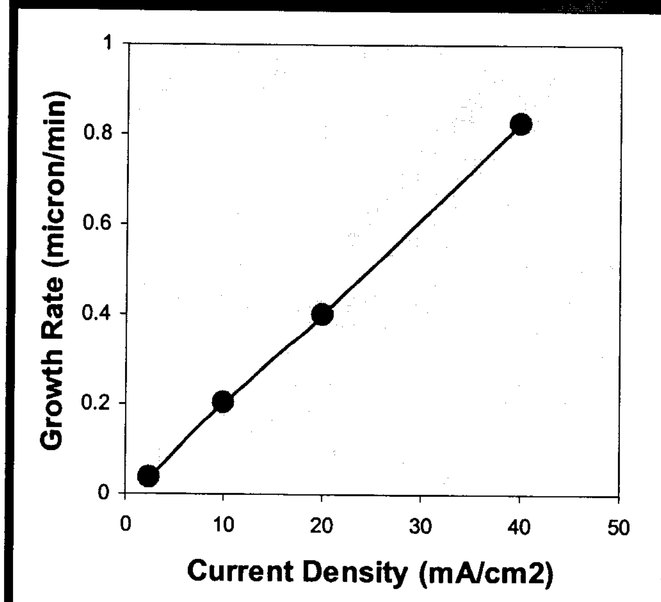




**SEM micrographs of electroplated Cu**



# Stress in Electroplated Ni

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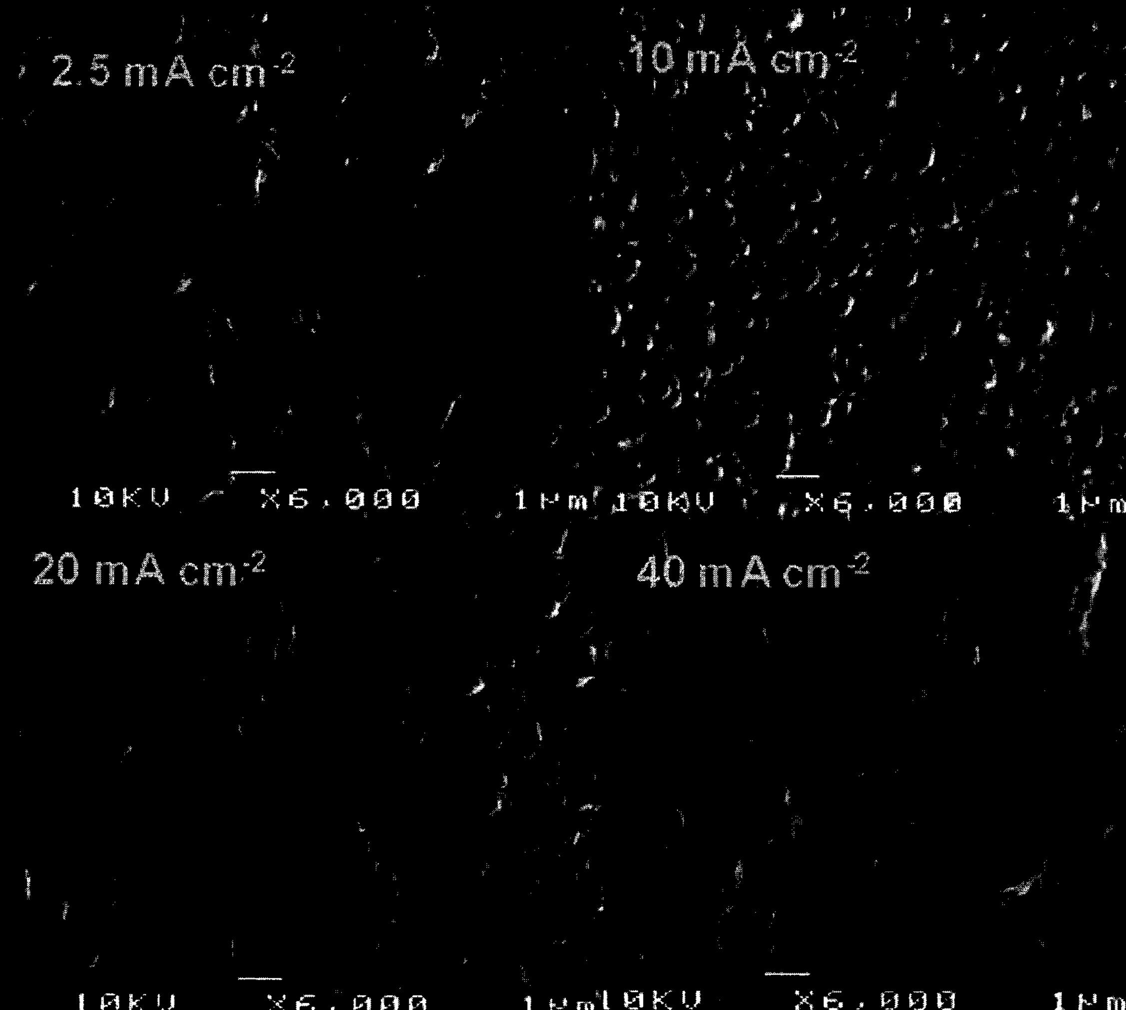
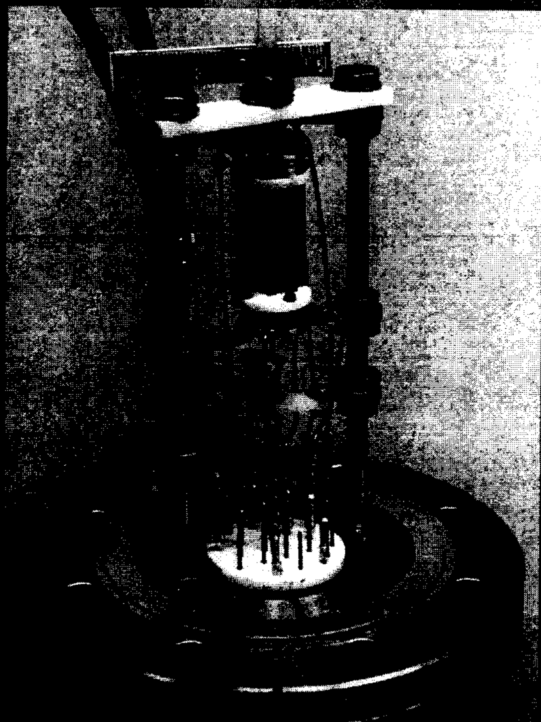
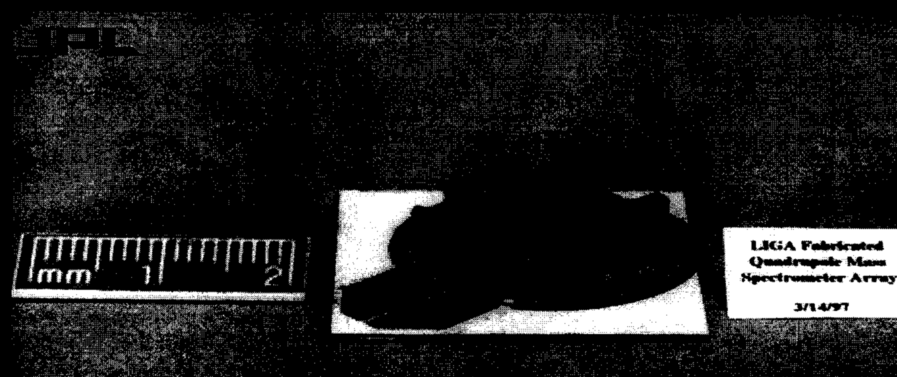


# JPL Fabricated Miniaturized Quadrupole Mass Filters



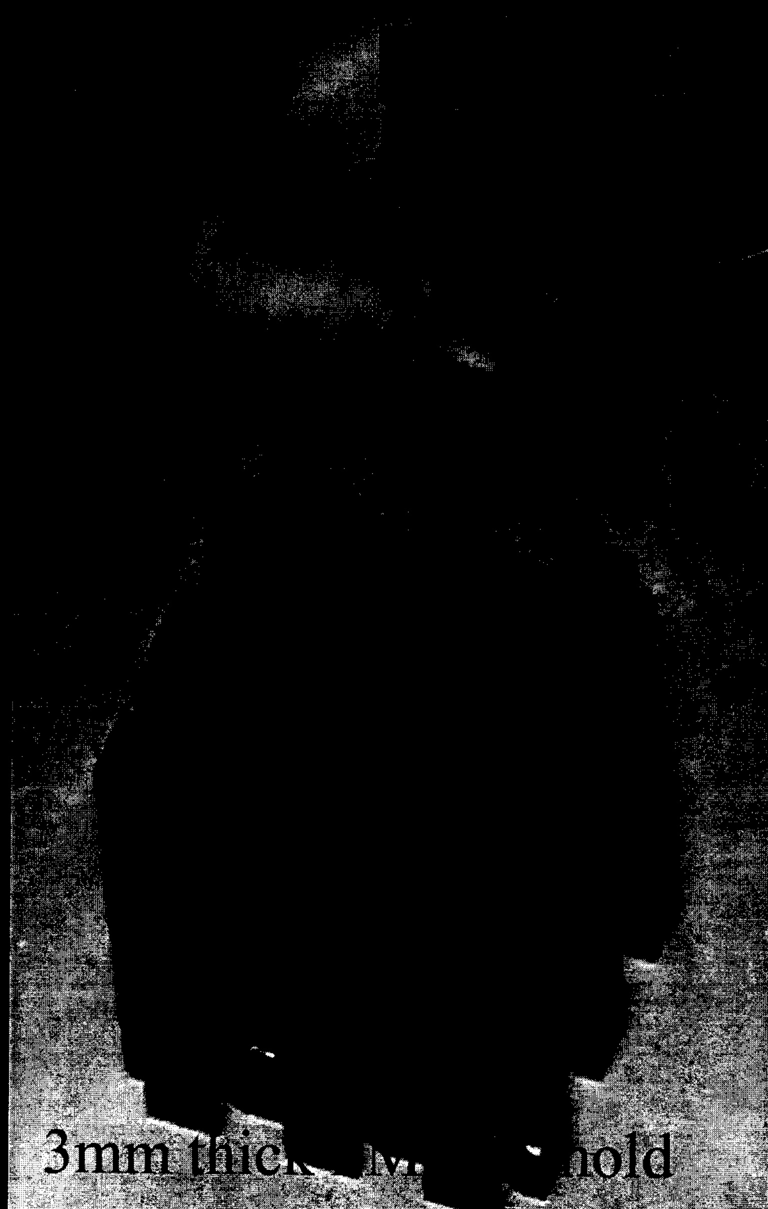
•<u>Conventional Machining</u>
•Approximately 25 mm pole length
•16 poles, 9 Quadrupoles
•2 dimensional array
(Chutjian, Orient, et al)

•<u>Electro-Discharge Machining (EDM)</u>
•Approximately 7 mm pole length
•4 poles, 1 Quadrupole
(Fuerstenau, Chutjian, Orient, et al)




•<u>LIGA Micromachining</u>
•Approximately 3 mm pole length
•20 poles, 9 Quadrupoles
•Linear array
(Wiberg, Chutjian, Orient, et al)

# LIGA Micromachined 2-D Quadrupole Arrays



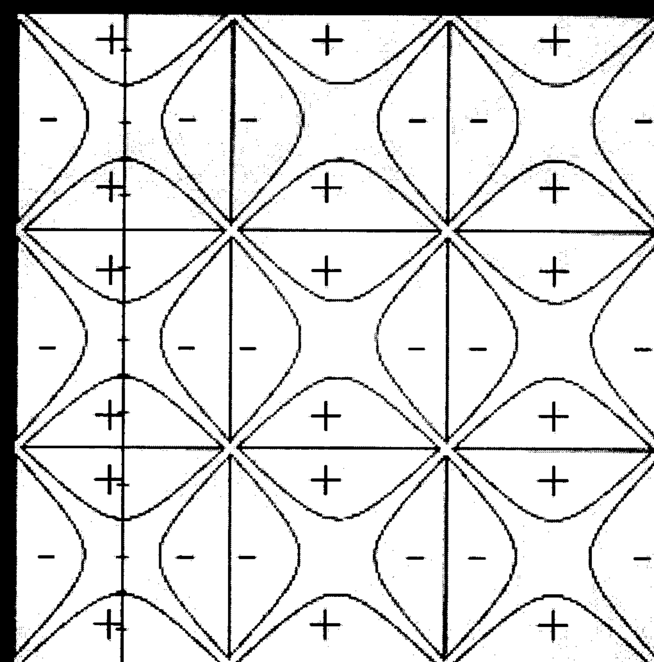
Material:

Non-magnetic Copper

Pole Length: 3 mm pole

# of poles : 24 poles

# of quadrupole: 9 Quadrupole

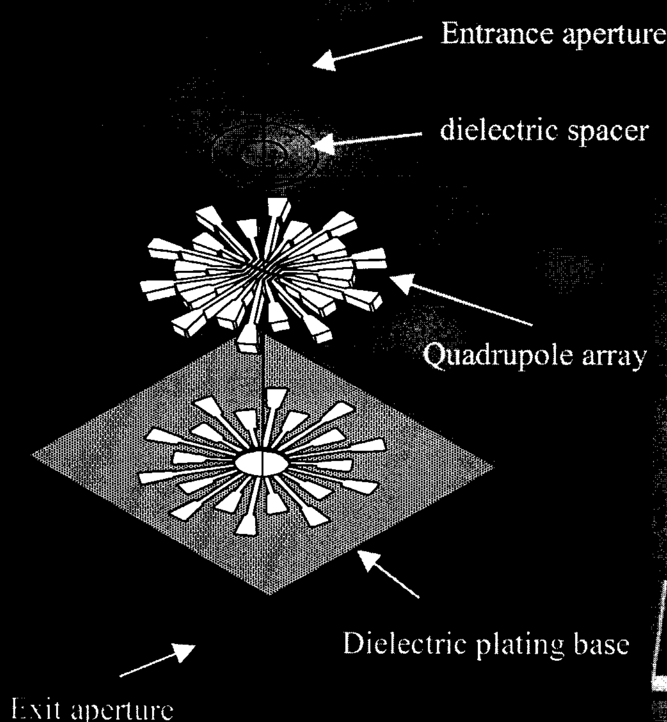


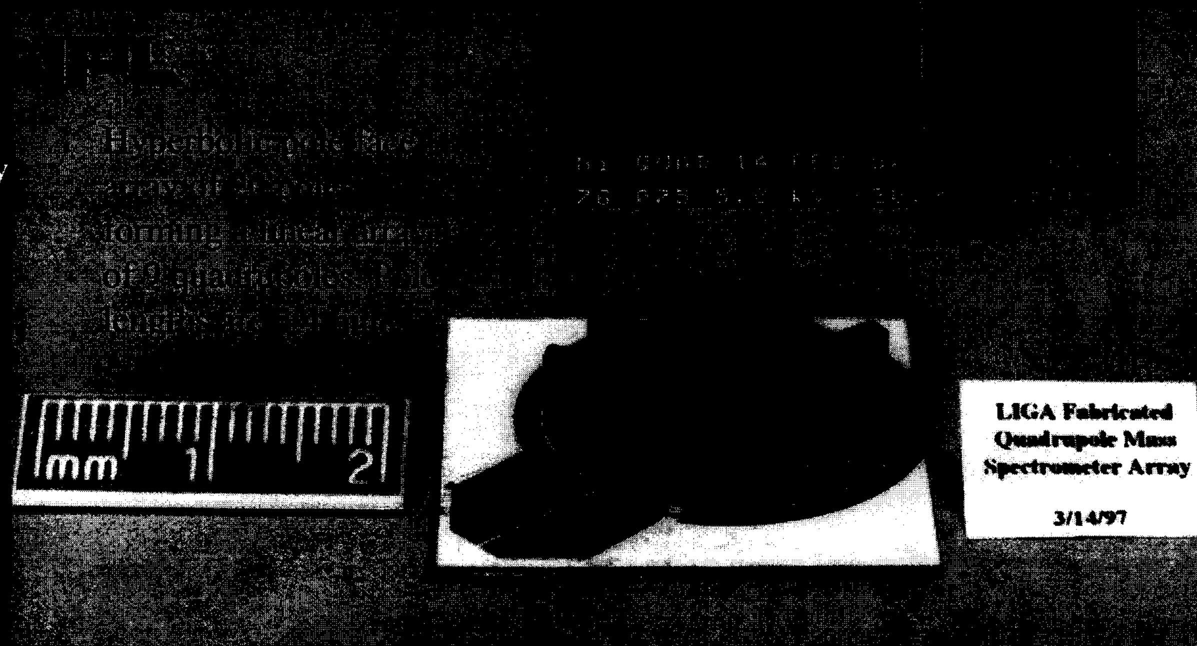
# LIGA Micromachined 2-D Quadrupole Arrays

# LIGA Fabricated 3 X 3 Arrays in 3” wafer

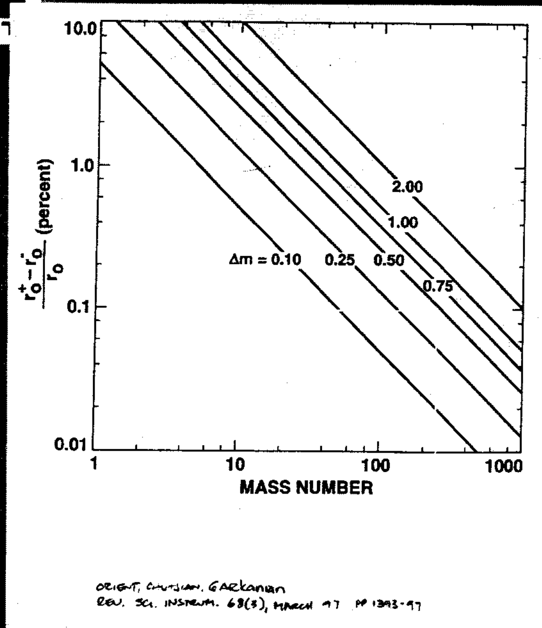
# LIGA Fabricated Linear Quadrupole Array

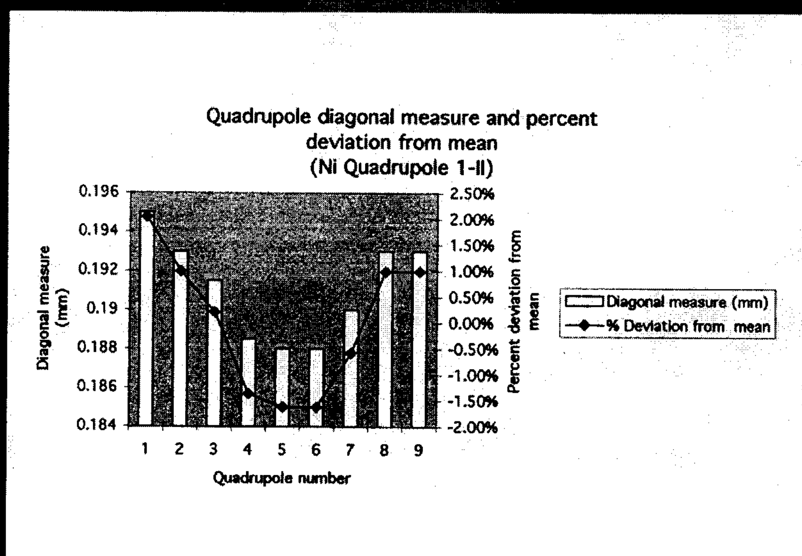
## Quadrupole Mass Filter

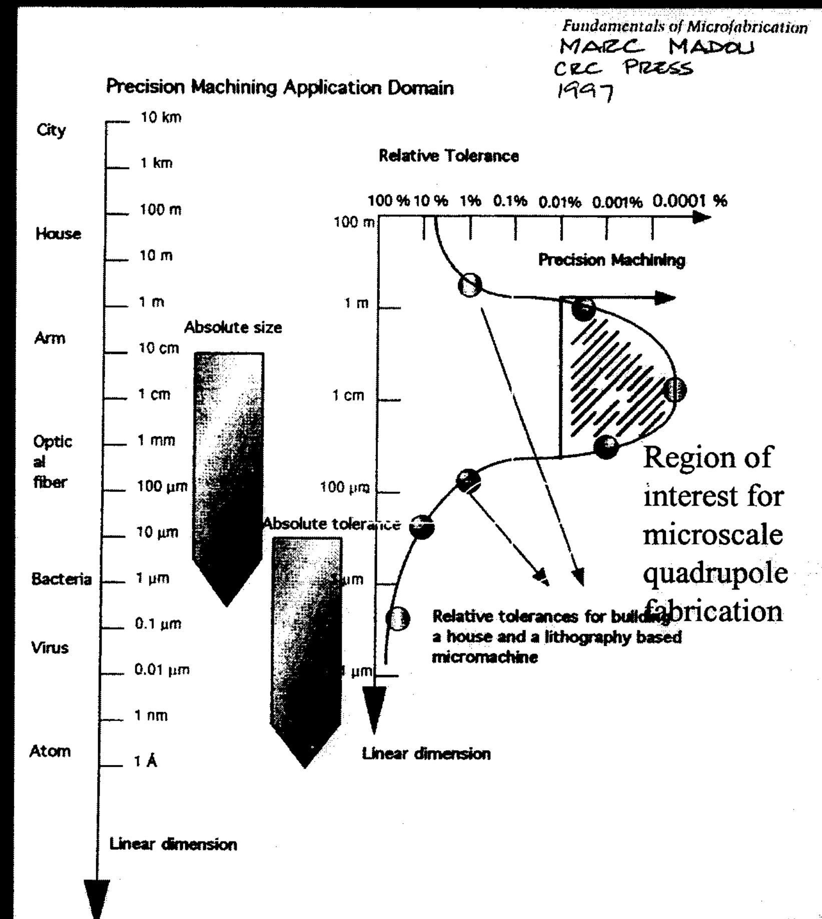


# Relative Precision Requirements for Quadrupole Fabrication



# Acknowledgements


**Funding Support (Current and Previous):**

| Project | Source | Program Manager |
|---|---|---|
| •GC/MS System | Code S | Tim Krabach |
| •Ion Traps | Code S | Neville Marzwell |
| •Scroll Pump | Code S | Neville Marzwell |
| •3D LIGA | JPL DRDF | |
| •LIGA Development | Code S | Virendra Sarohia |
| •Quadrupole | PIDP | |

**Contributors:**

| Element | Affiliation | PI |
|---|---|---|
| •Piezo Check Valves | Boston University | Jan Smits |
| •Turbo Pump (SBIR) | Creare inc. | Robert Kline-Schroder |
| •Minature GC (SBIR) | Thorleaf Research inc. | Paul Holland |
| •Turbo Pump (SBIR) | Pheonix Analysis and Design | Mark Johnson |
| •Atmosheric Analysis | Howard University | Vernon Morris |
| •Scroll Pump Design | Lawrence Berkeley Nat. Lab. | Keith Jackson |
| •LIGA Fabrication | Sandia National Lab. | Jill Hruby |

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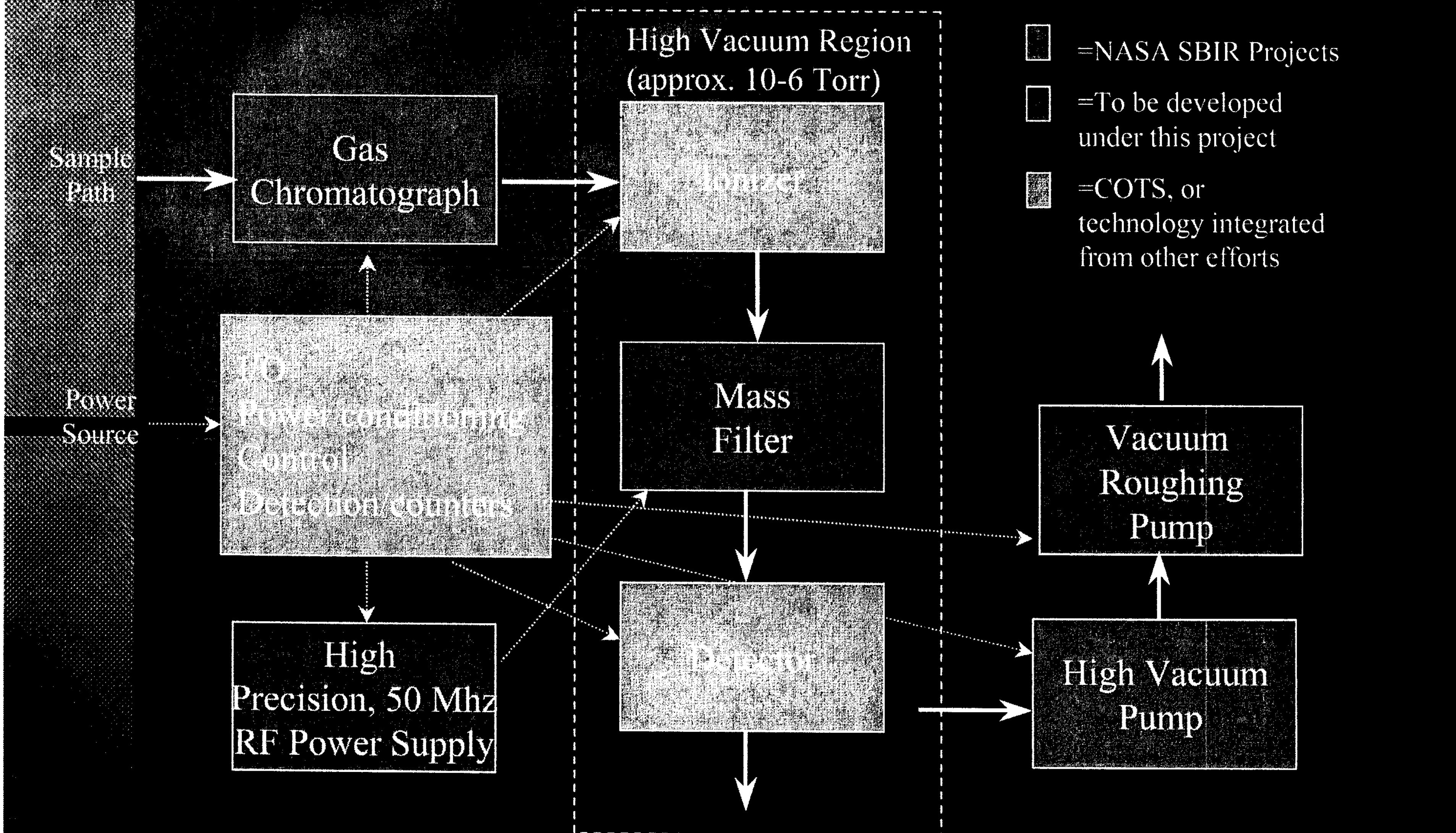

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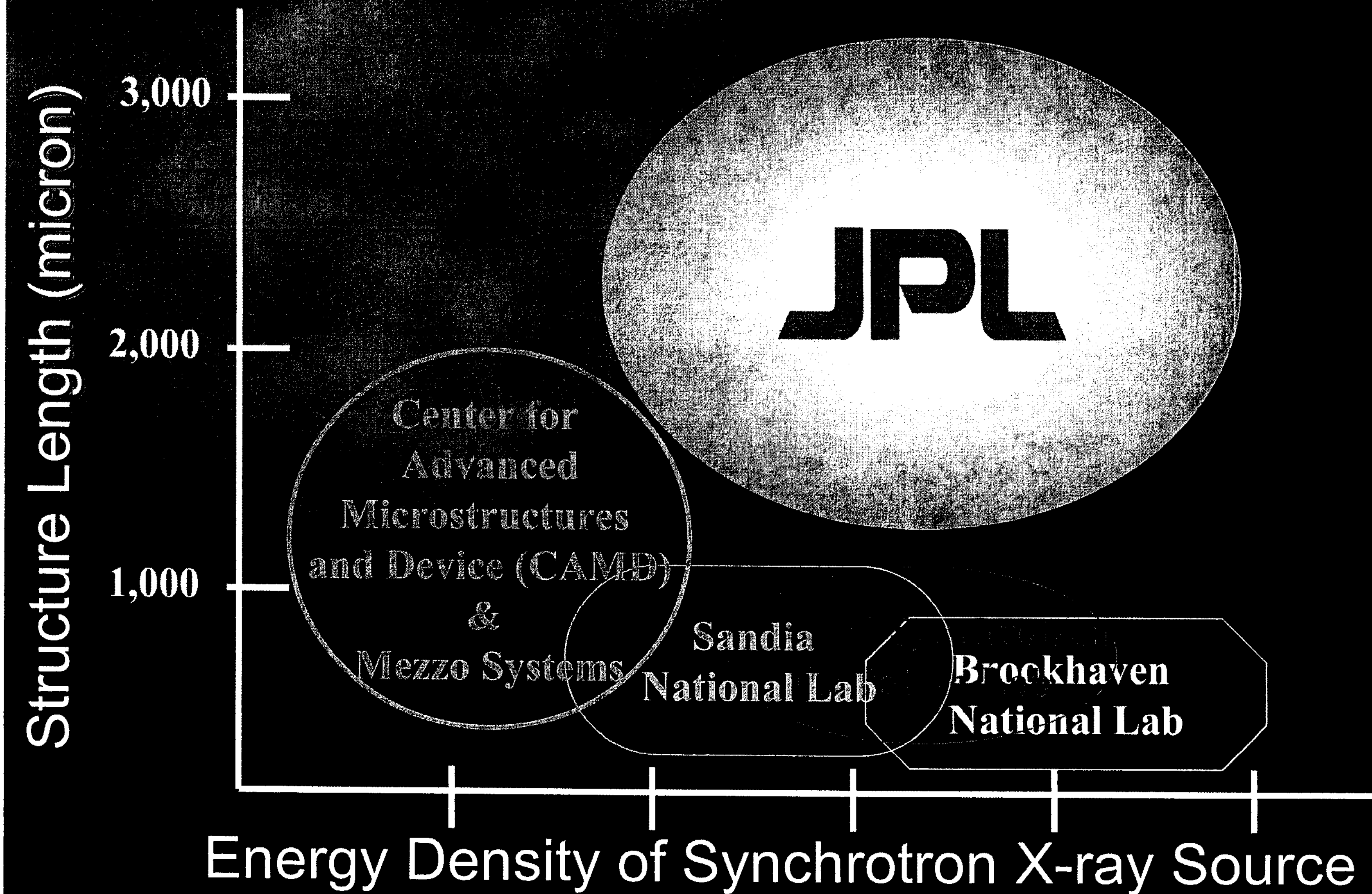

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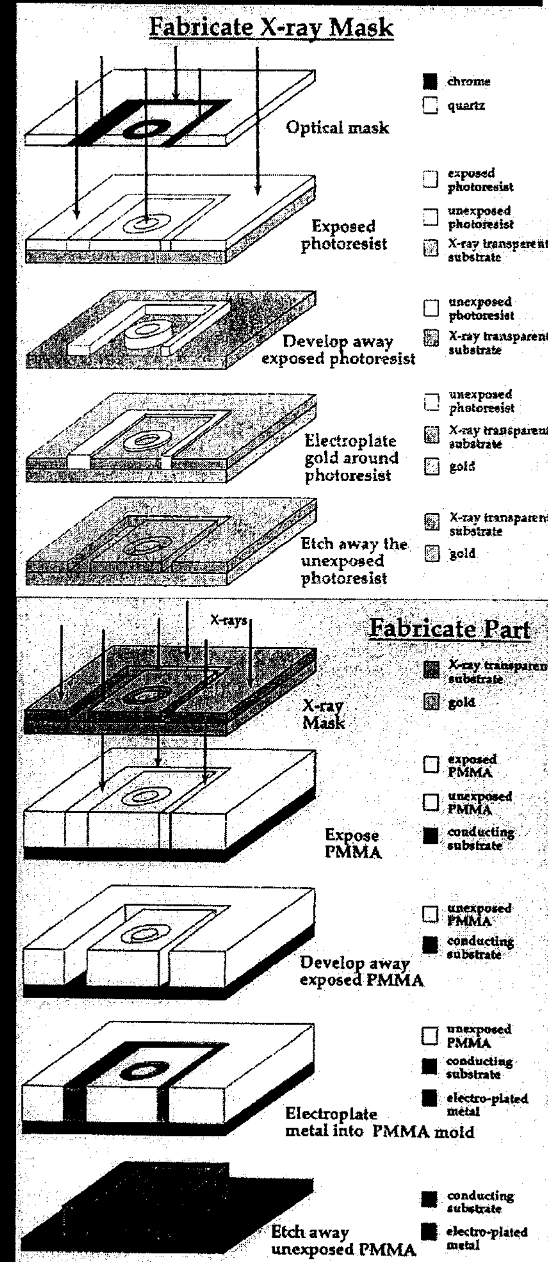

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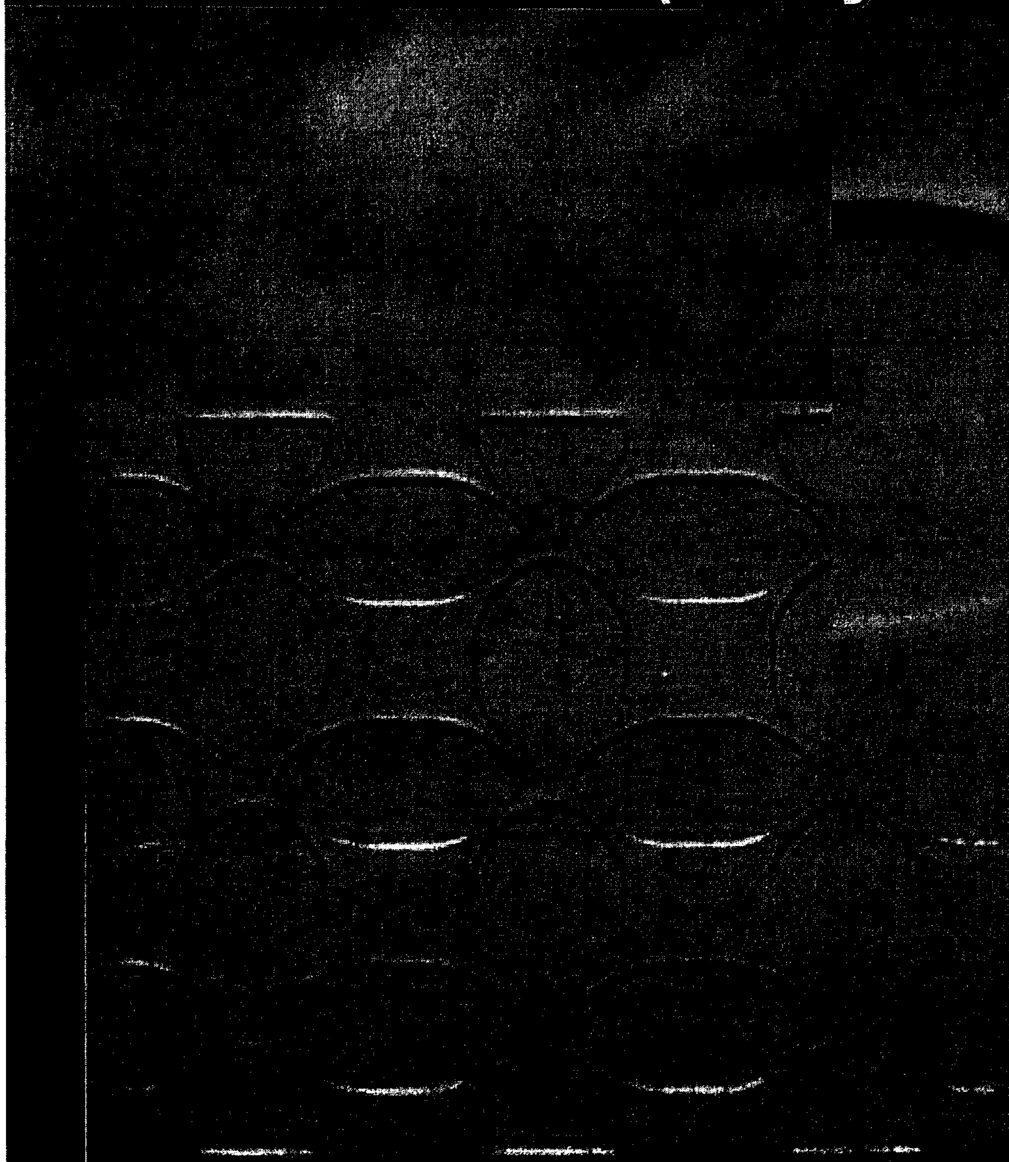

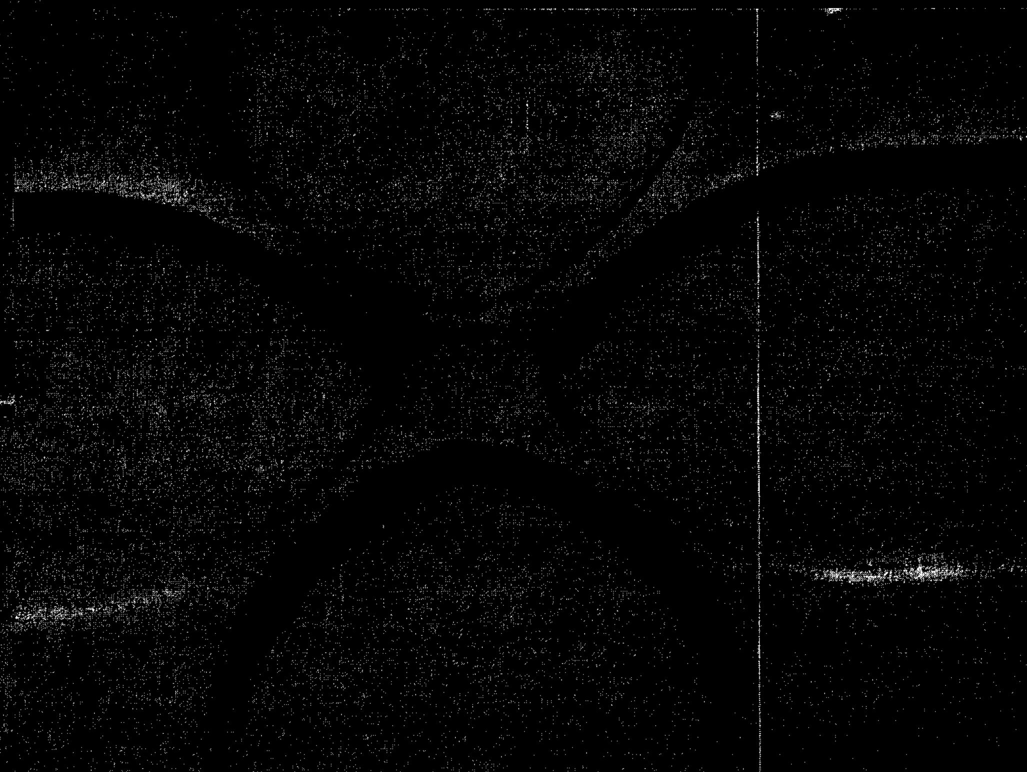

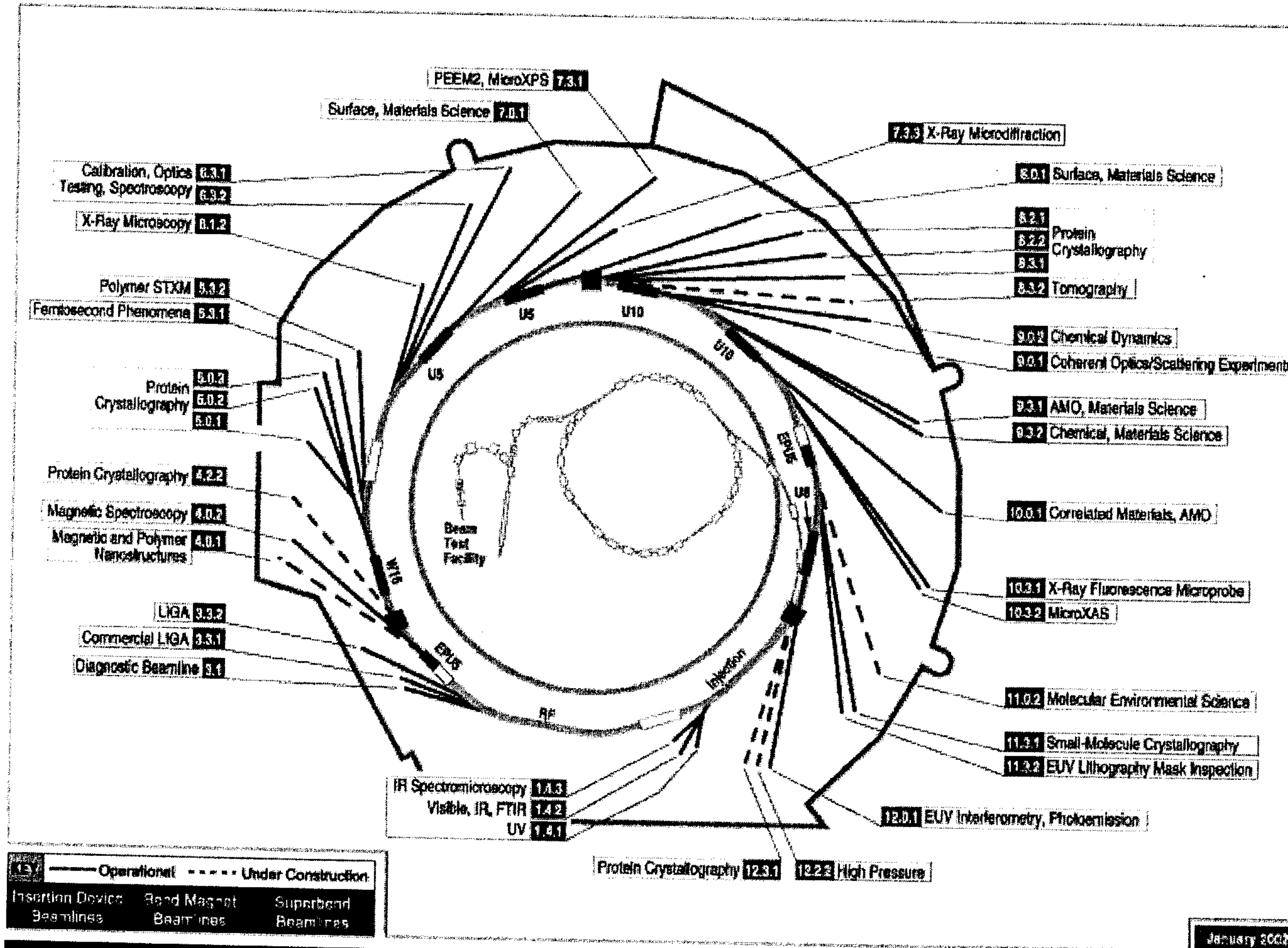


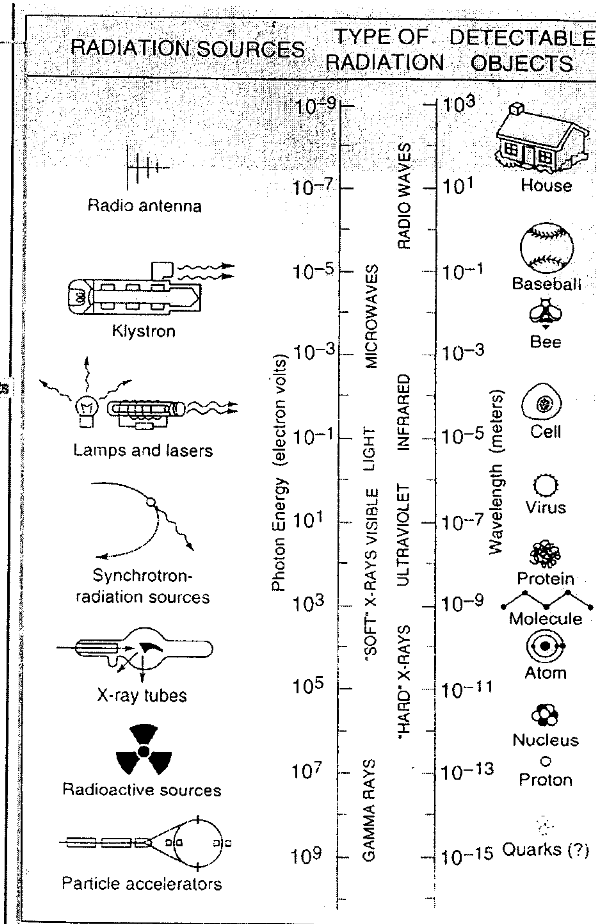


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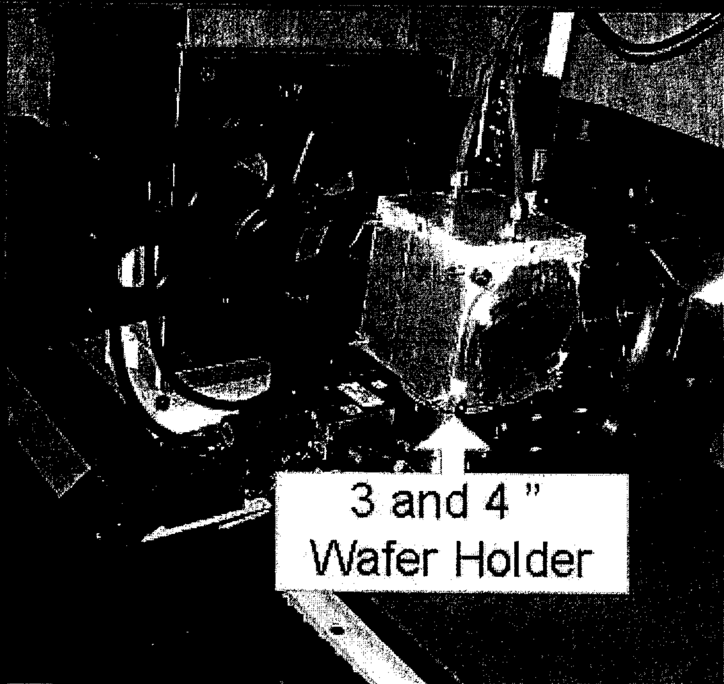
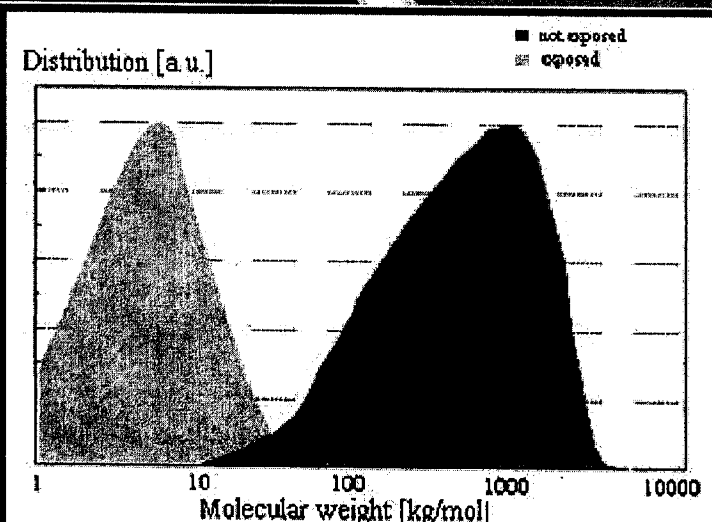
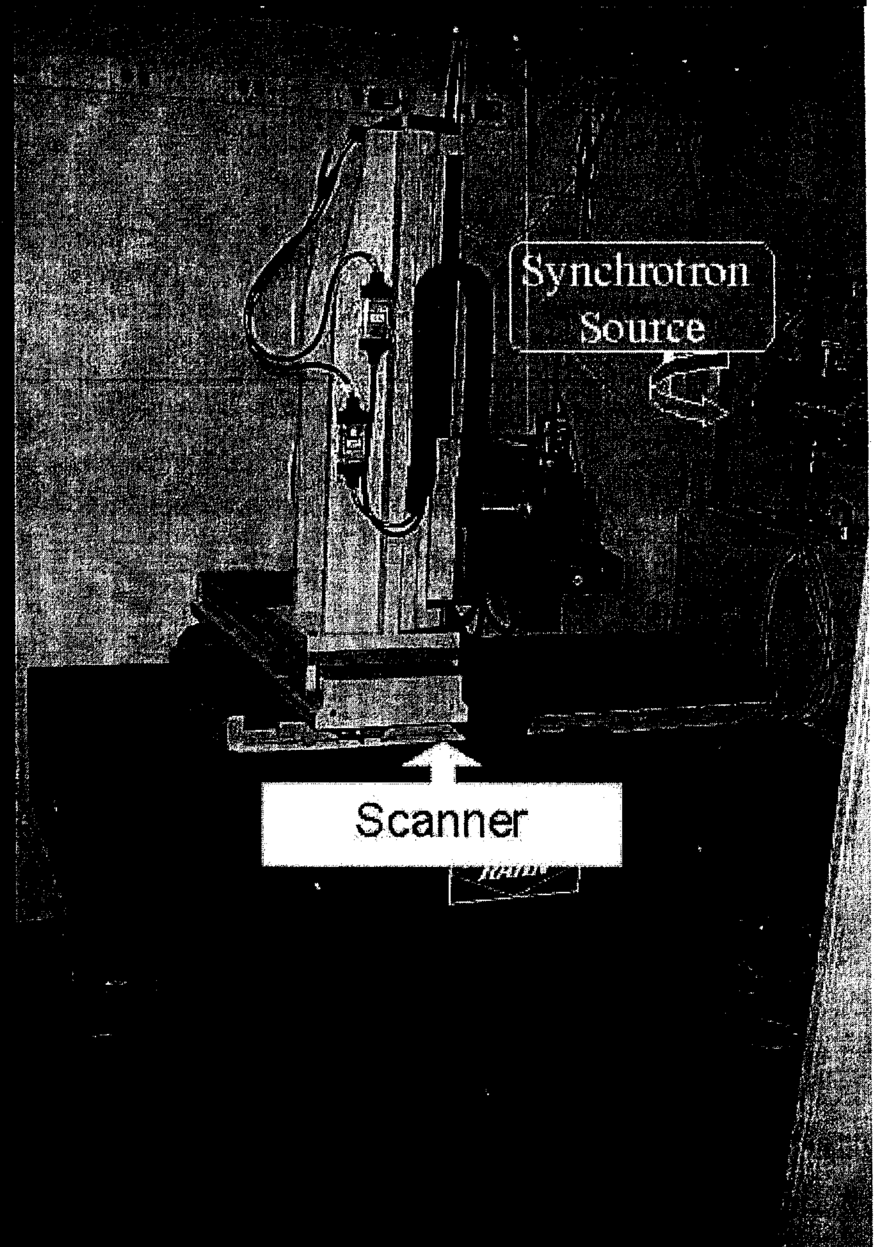
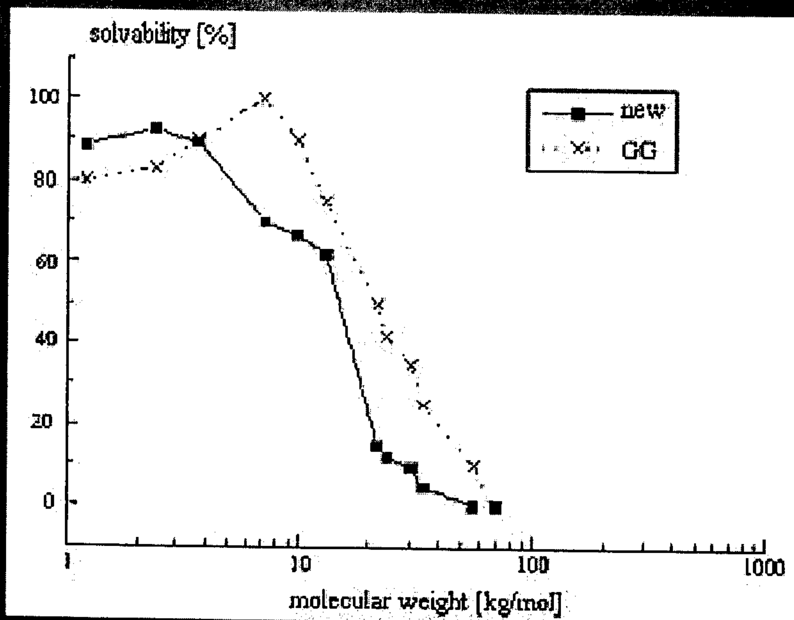


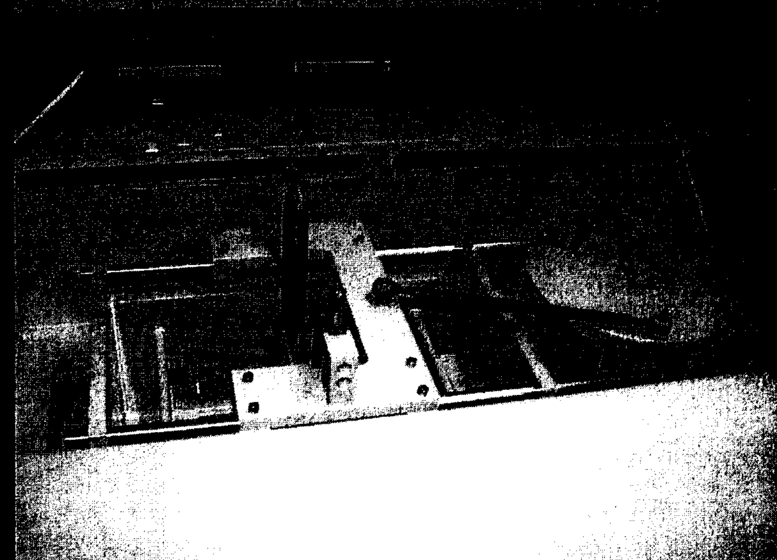
# Developing



solvability [%]
100
80
60
40
20
0
new
GG
1
10
100
1000
molecular weight [kg/mol]

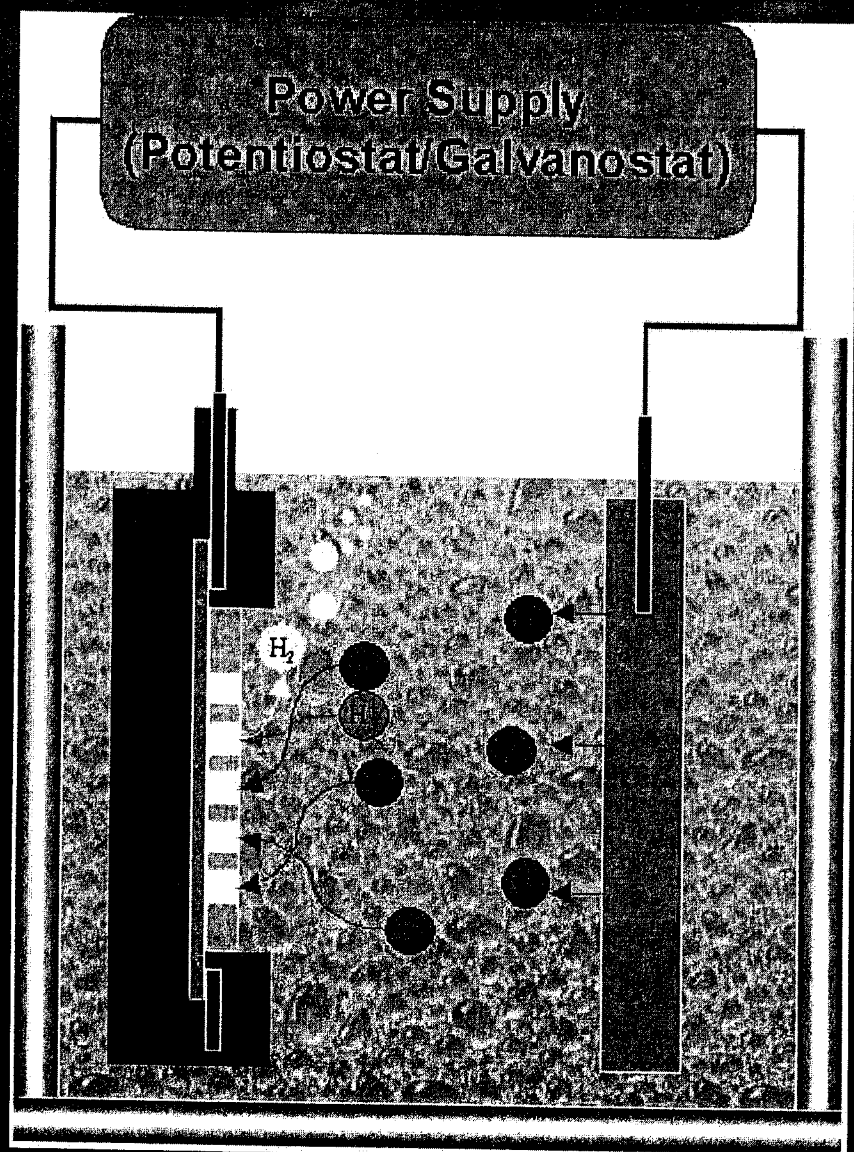
# Electroplating

Electroplating Stations with fine temperature and process control



Electroplating Paddle Cell

# Electroplating apparatus




Cathodic(Reduction) Reaction

e.g.

$Ni^{+2} + 2e^- \rightarrow Ni_{(s)}$

$2H^+ + 2e^- \rightarrow H_2$ (side reaction)

Anodic(Oxidation) Reaction

e.g.

$Ni_{(s)} \rightarrow Ni^{+2} + 2e^-$

Anode

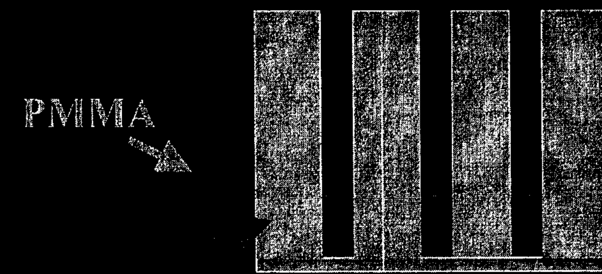
Substrate

PMMA (mold)

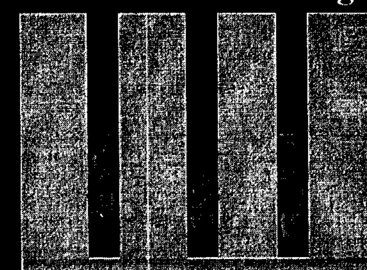
Substrate holder

# Electroplating

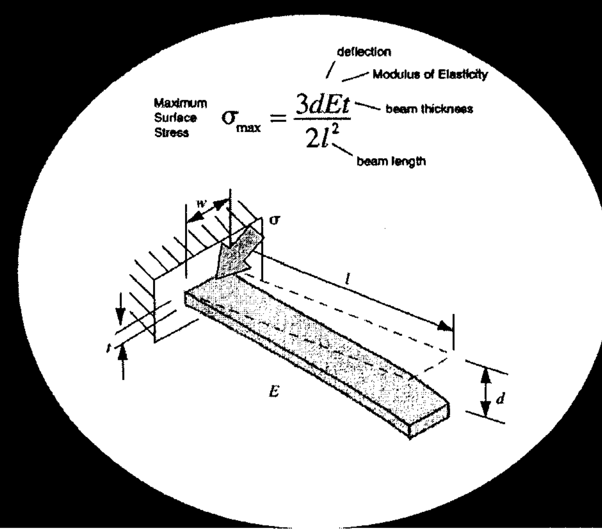
- Developing new electroplating solution to enhance mass transfer in deep trench and promote uniform growth (eliminate dendrite and powdery growth)
- Developing electroplating solution and processes to minimize internal stress in the deposits



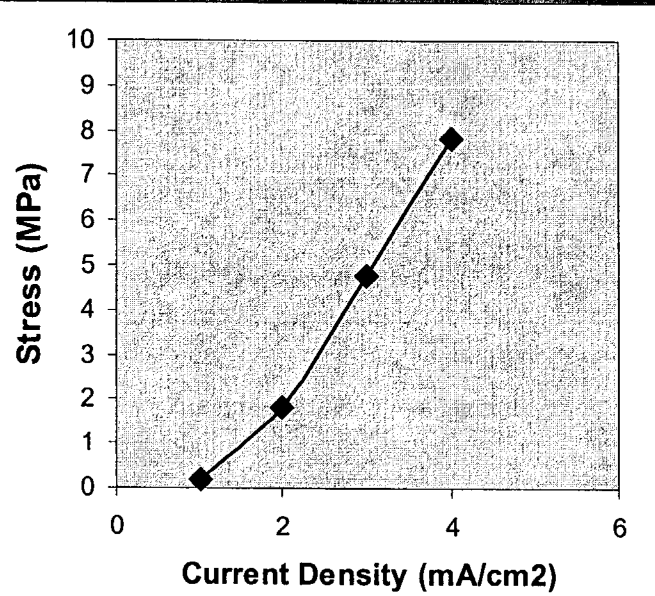

**Uniform 2-dimensional growth**

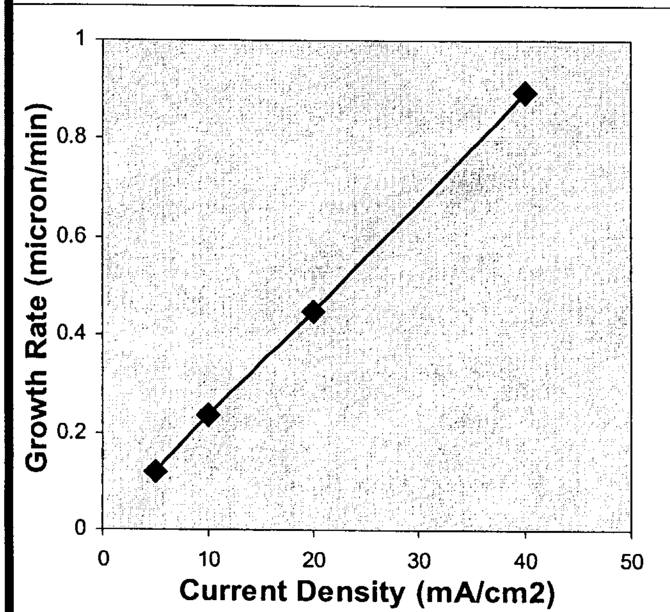


**Non-uniform dendrite or powdery growth**

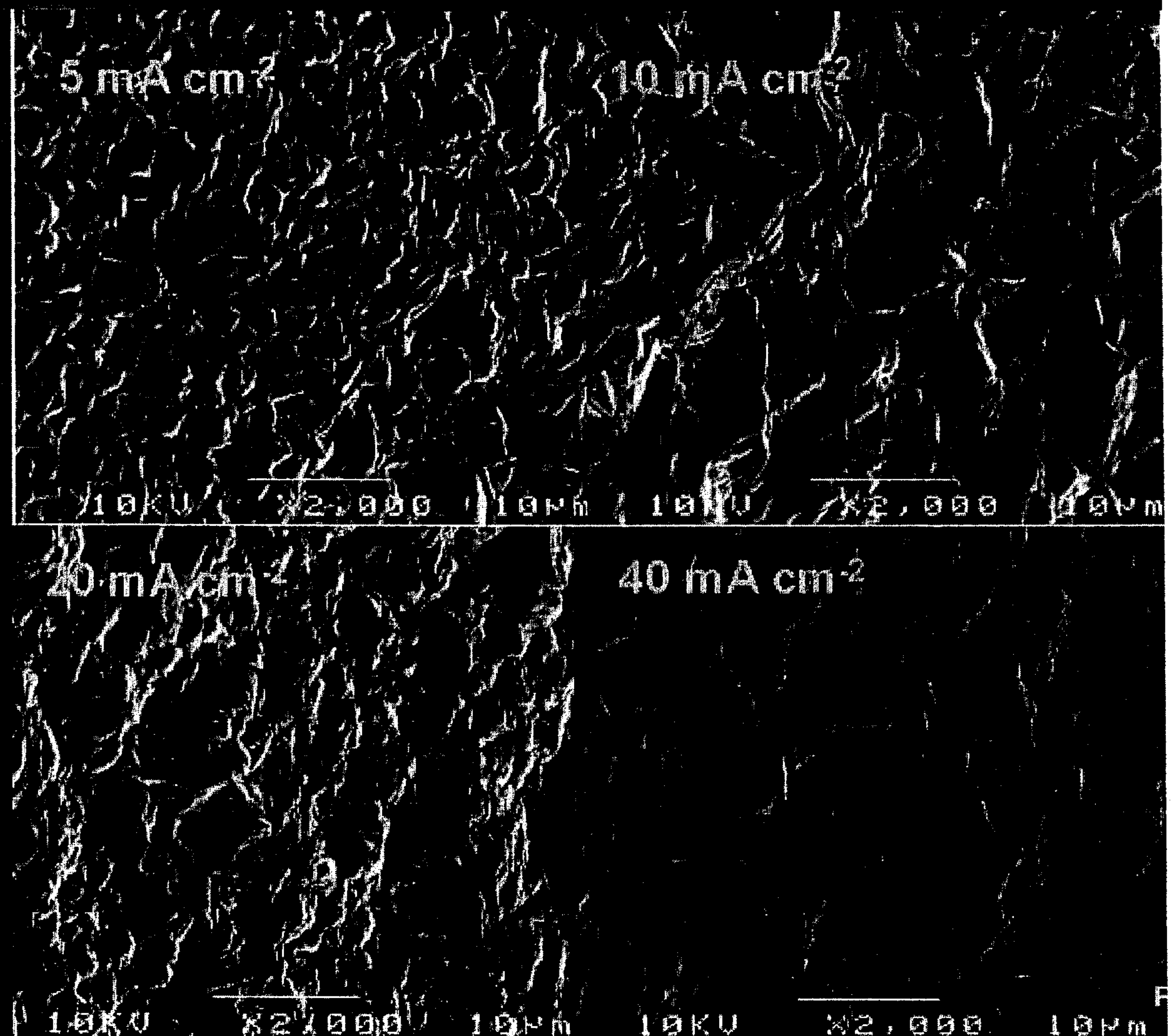


# Stress in Electroplated Cu

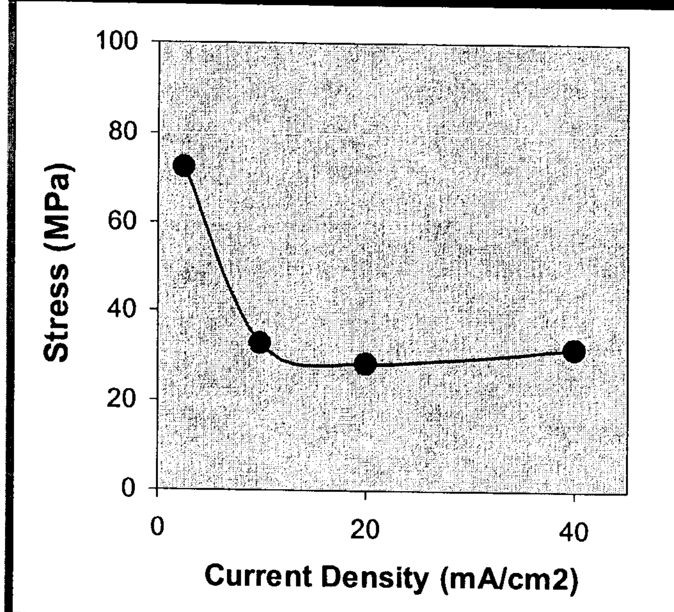


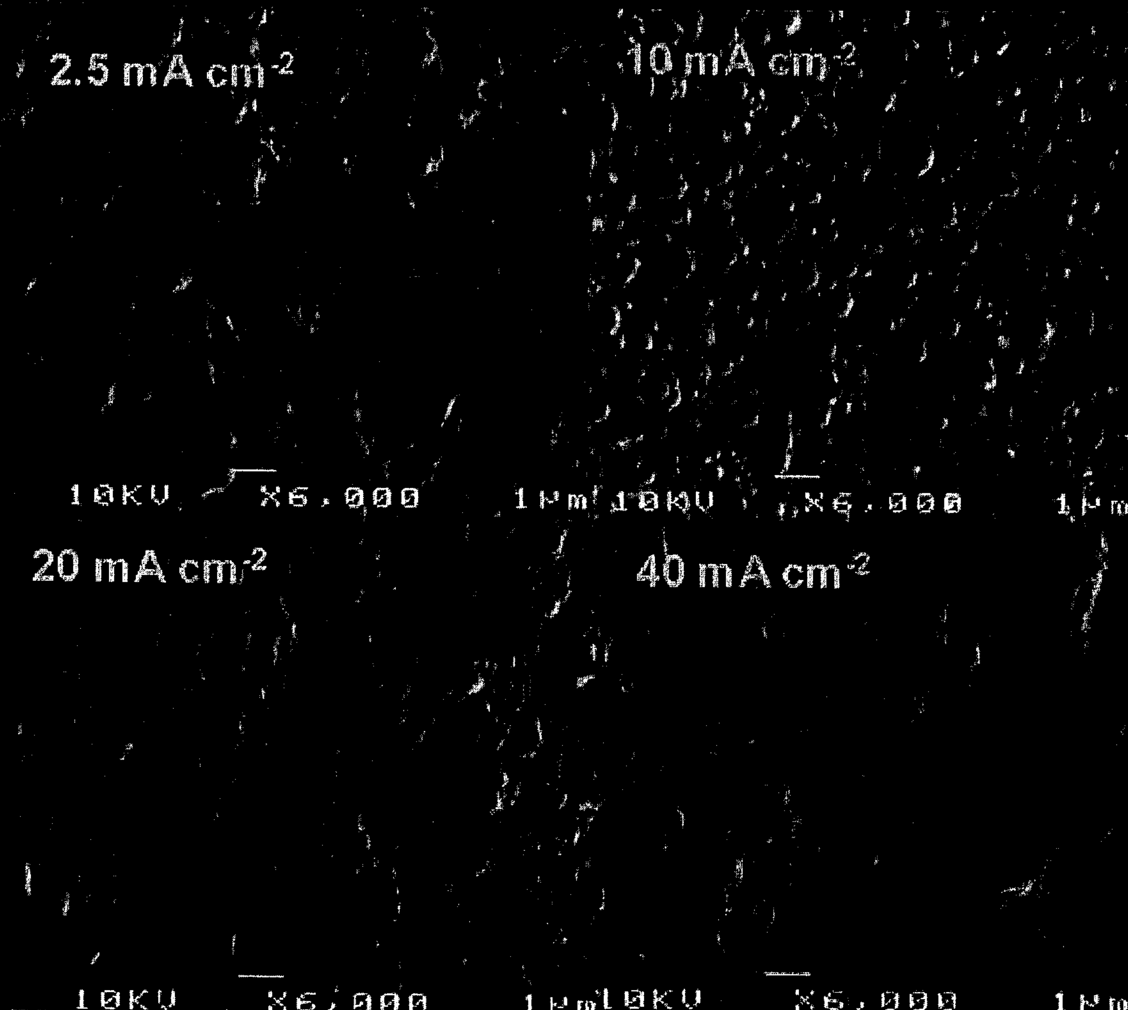

## SEM micrographs of electroplated Cu

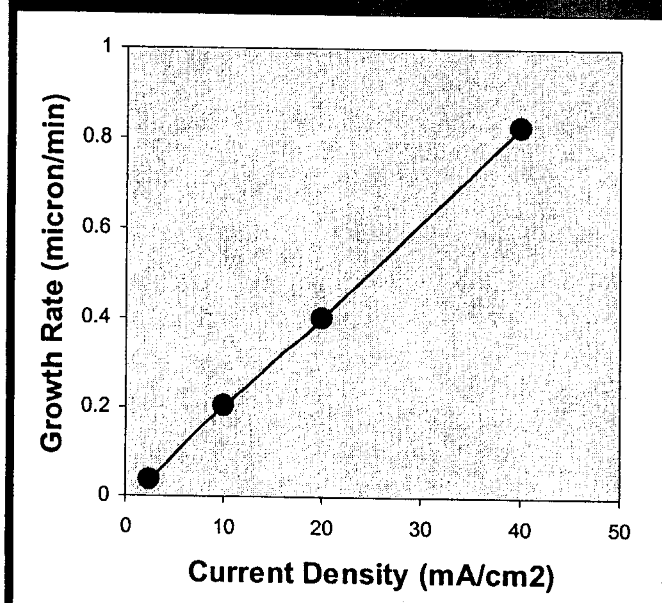


# Stress in Electroplated Ni

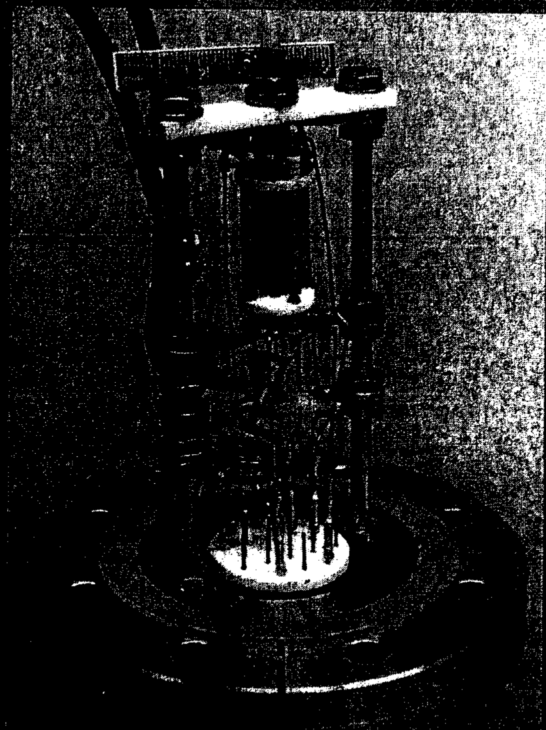



## SEM micrographs of electroplated Ni



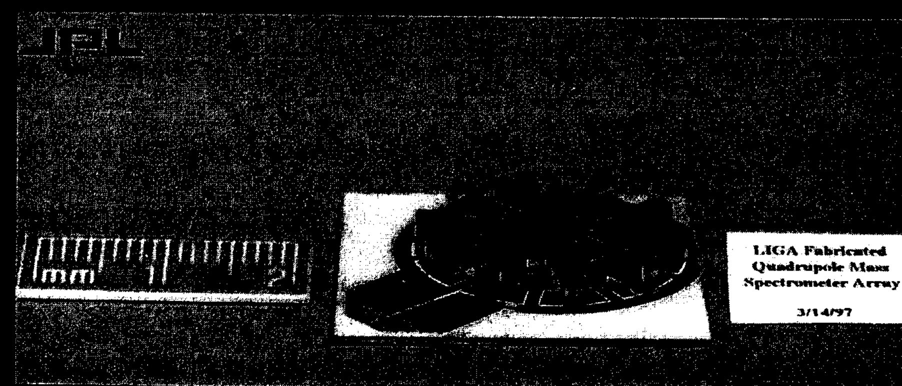



# JPL Fabricated Miniaturized Quadrupole Mass Filters



•Conventional Machining
•Approximately 25 mm pole length
•16 poles, 9 Quadrupoles
•2 dimensional array
(Chutjian, Orient, et al)

•Electro-Discharge Machining (EDM)
•Approximately 7 mm pole length
•4 poles, 1 Quadrupole
(Fuerstenau, Chutjian, Orient, et al)




•LIGA Micromachining
•Approximately 3 mm pole length
•20 poles, 9 Quadrupoles
•Linear array
(Wiberg, Chutjian, Orient, et al)

# LIGA Micromachined 2-D Quadrupole Arrays

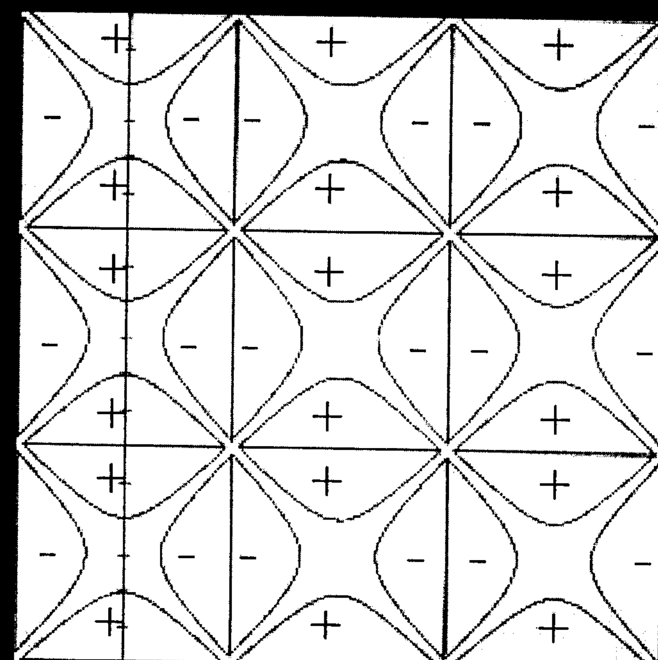
Material:

Non-magnetic Copper

Pole Length: 3 mm pole

# of poles : 24 poles

# of quadrupole: 9 Quadrupole



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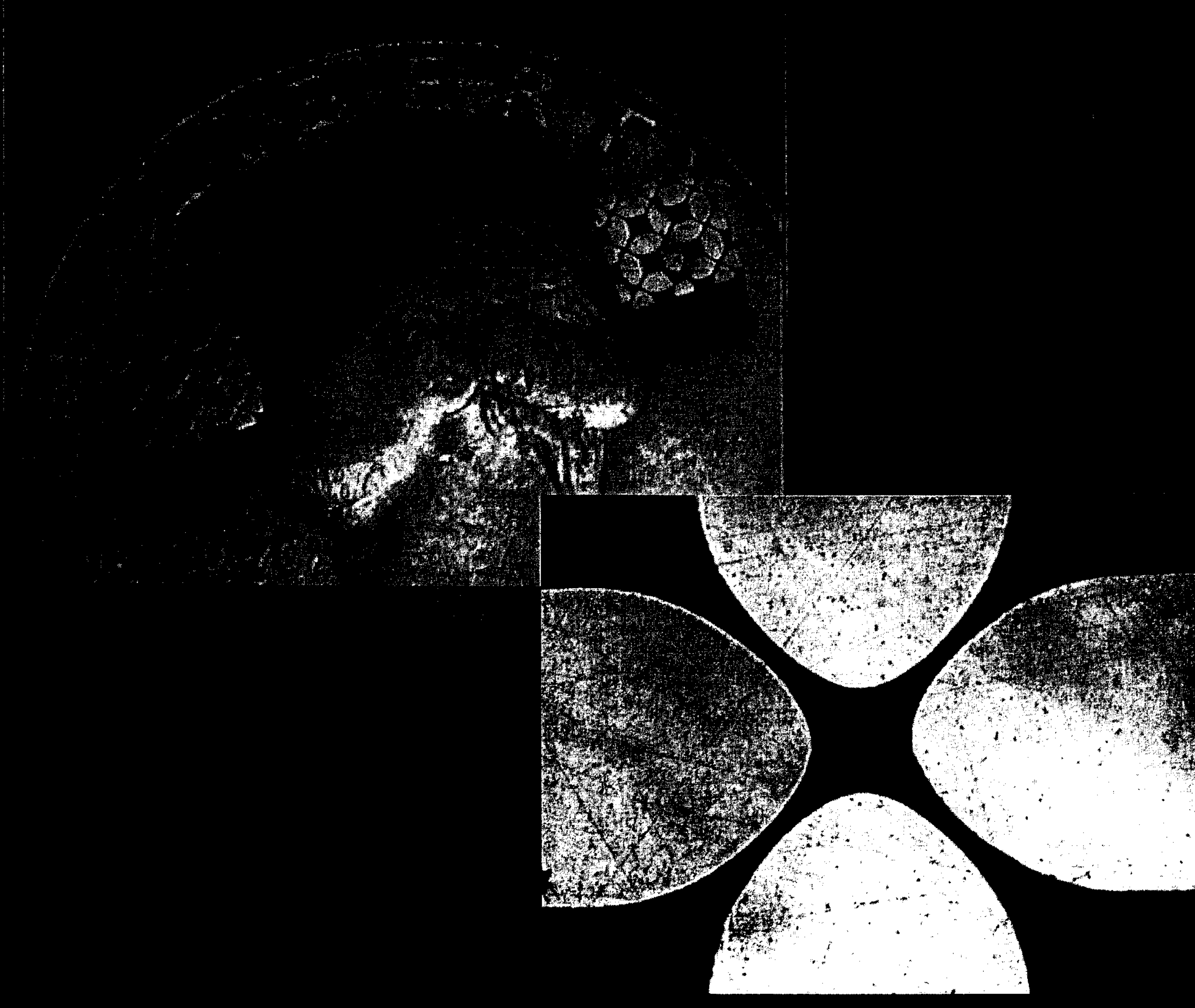
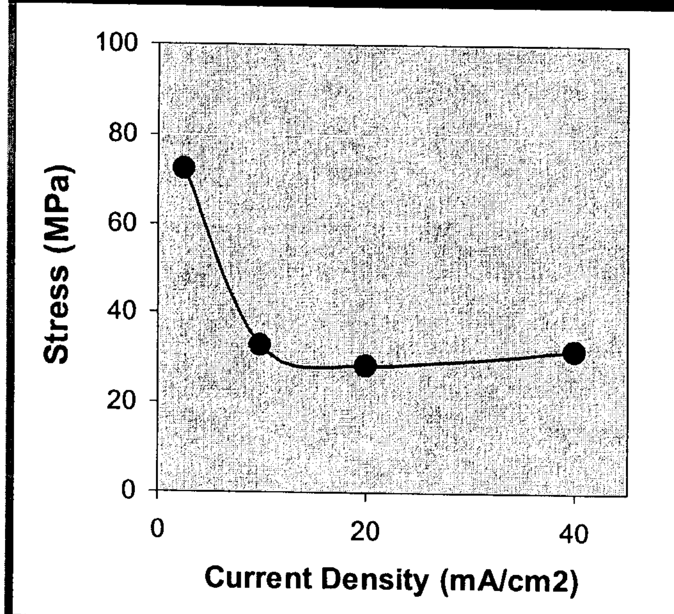
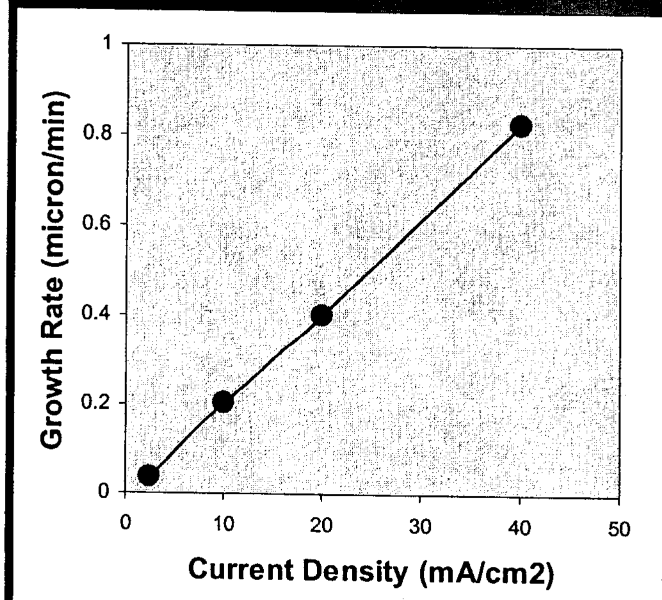
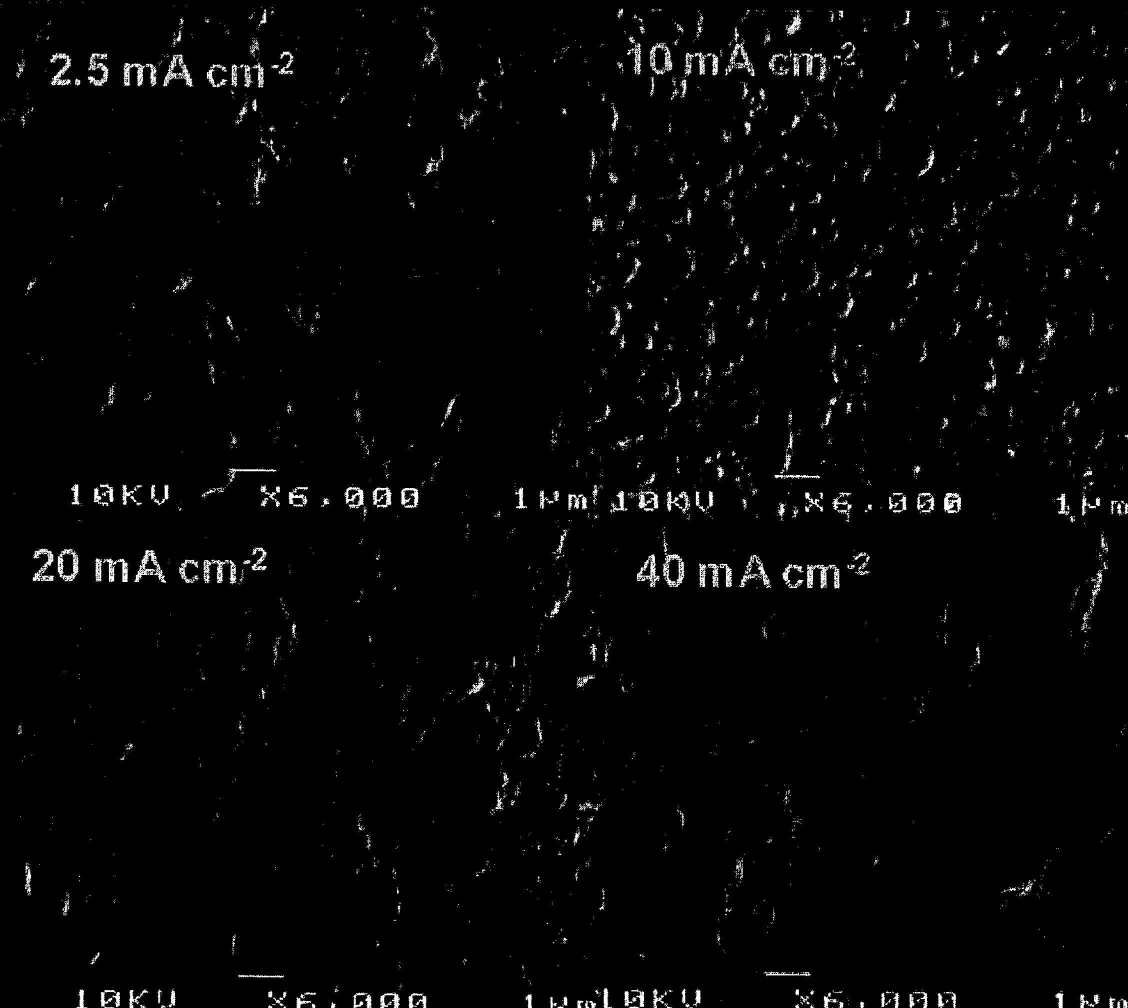


# Stress in Electroplated Ni

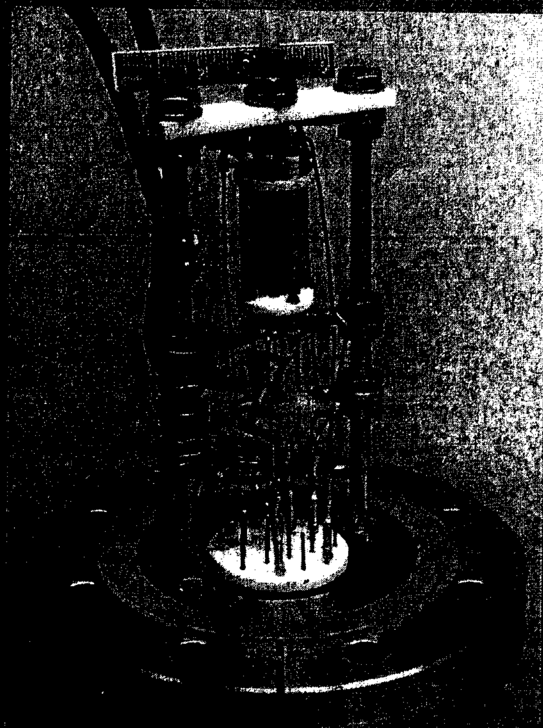



## SEM micrographs of electroplated Ni



# JPL Fabricated Miniaturized Quadrupole Mass Filters



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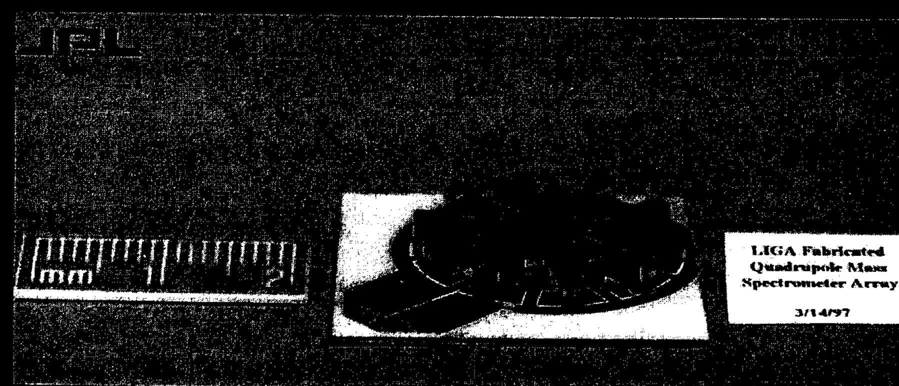


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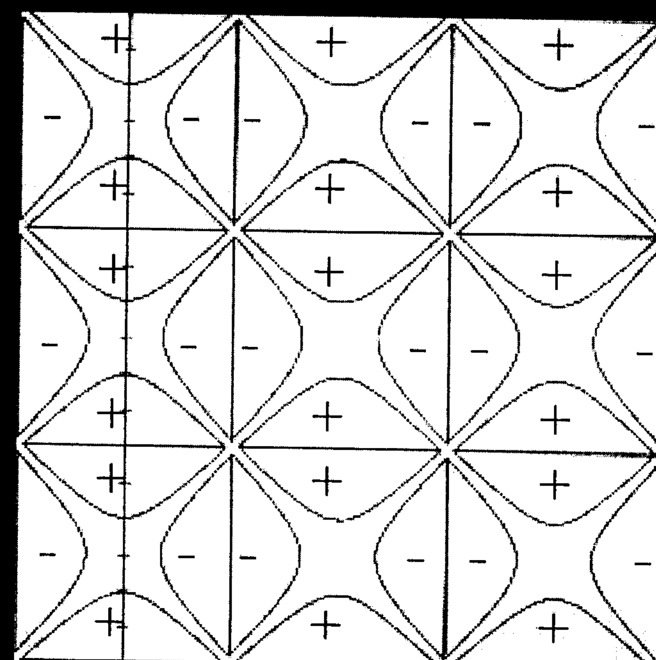
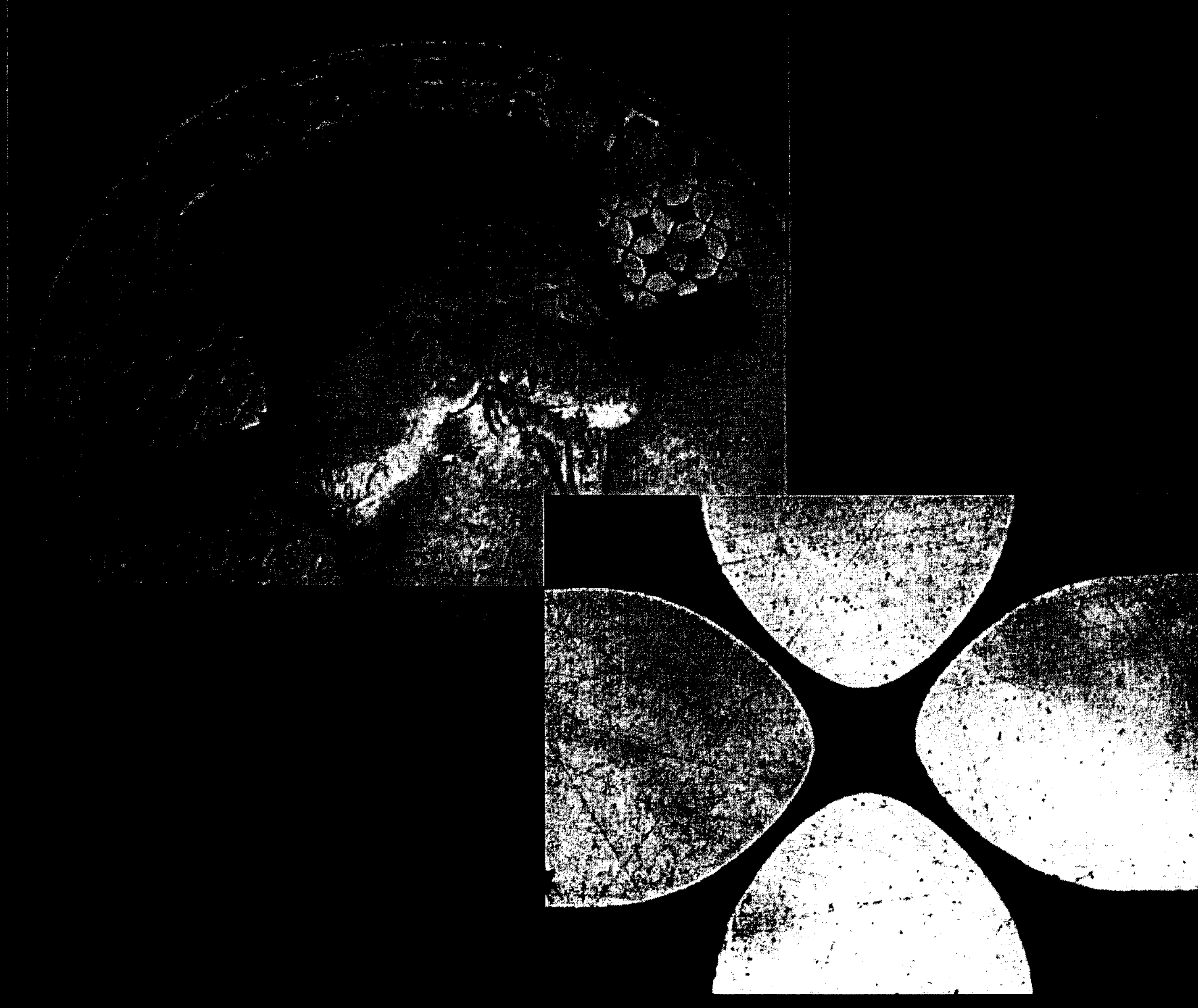


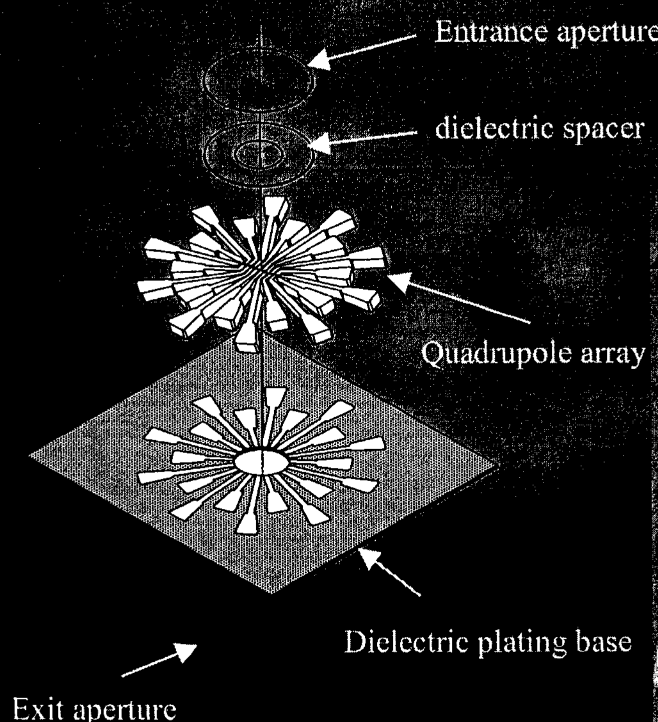
# LIGA Micromachined 2-D Quadrupole Arrays

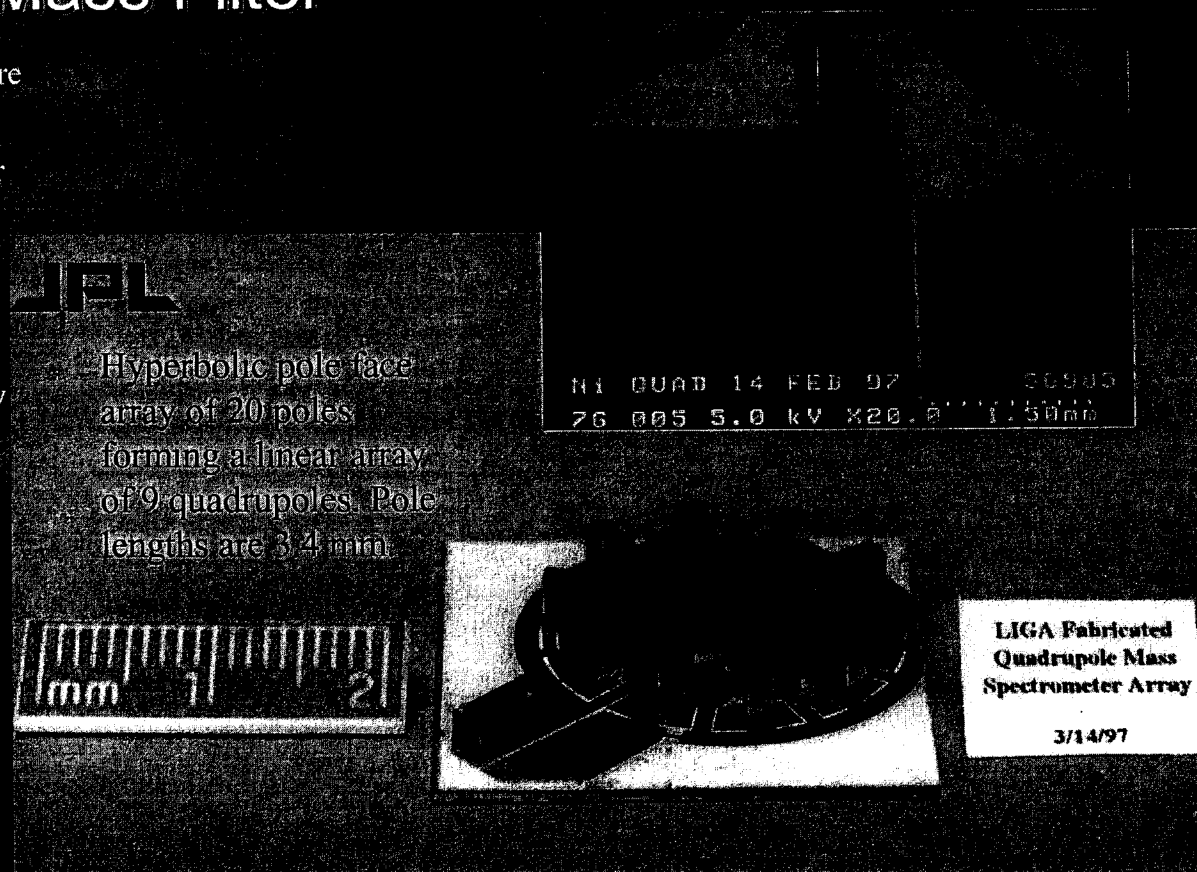


# LIGA Fabricated 3 X 3 Arrays in 3" wafer

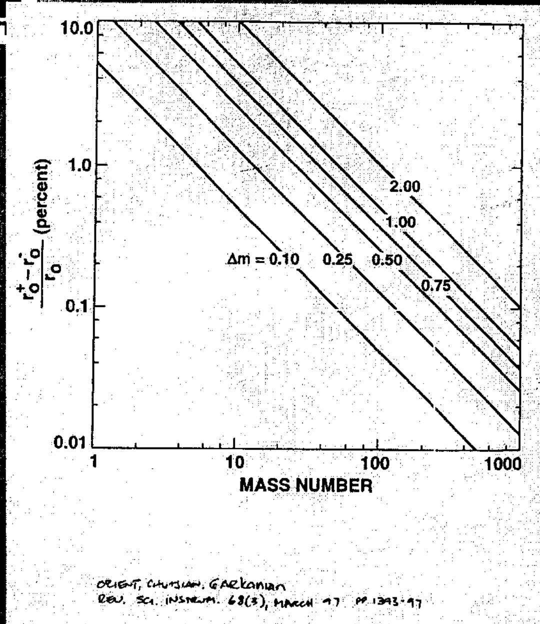
# LIGA Fabricated Linear Quadrupole Array

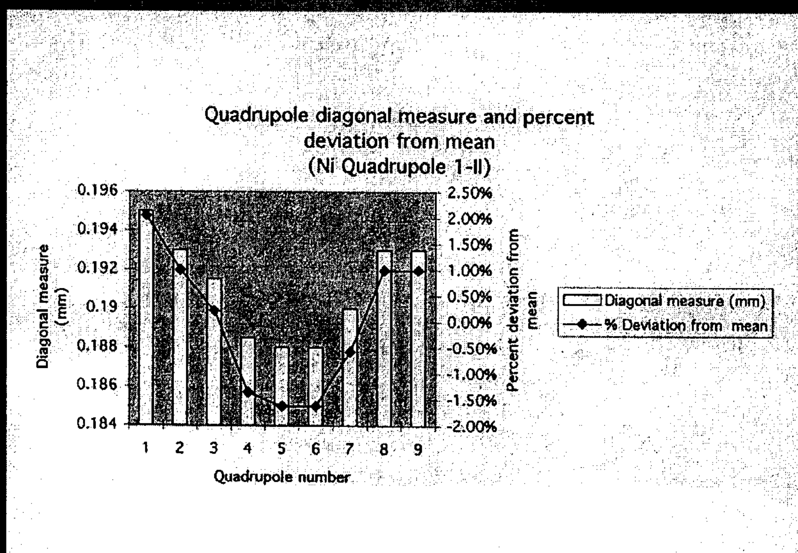
- Quadrupole Mass Filter

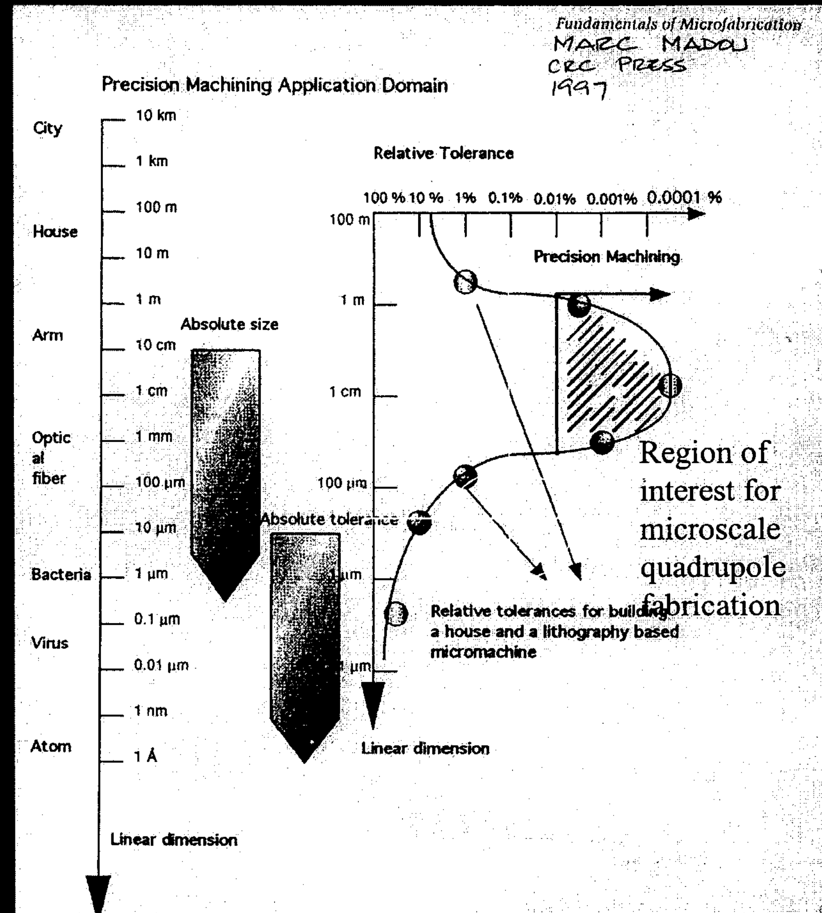


# Relative Precision Requirements for Quadrupole Fabrication



# Acknowledgements


**Funding Support (Current and Previous):**

| Project | Source | Program Manager |
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| •GC/MS System | Code S | Tim Krabach |
| •Ion Traps | Code S | Neville Marzwell |
| •Scroll Pump | Code S | Neville Marzwell |
| •3D LIGA | JPL DRDF | |
| •LIGA Development | Code S | Virendra Sarohia |
| •Quadrupole | PIDP | |

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| Element | Affiliation | PI |
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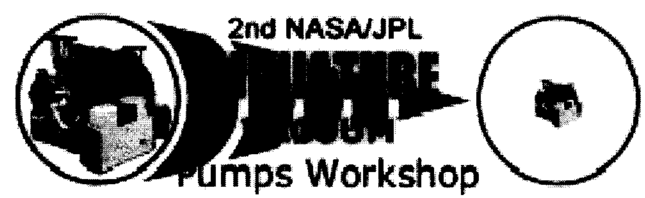
Ellie Trevarthen / HEMS 2002
Jet Propulsion Laboratory
4800 Oak Grove Drive, M/S 306-463
Pasadena, CA 91109-8099

# The 3rd Workshop on Harsh-Environment Mass Spectrometry

and the

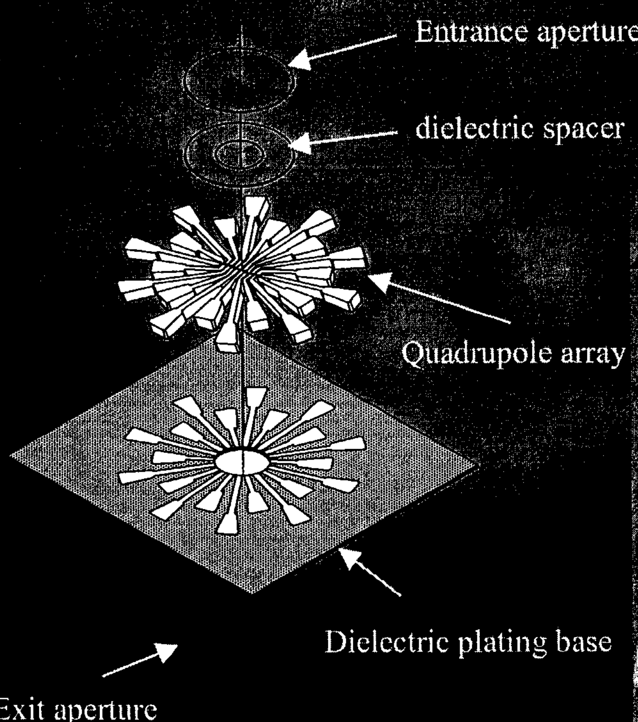


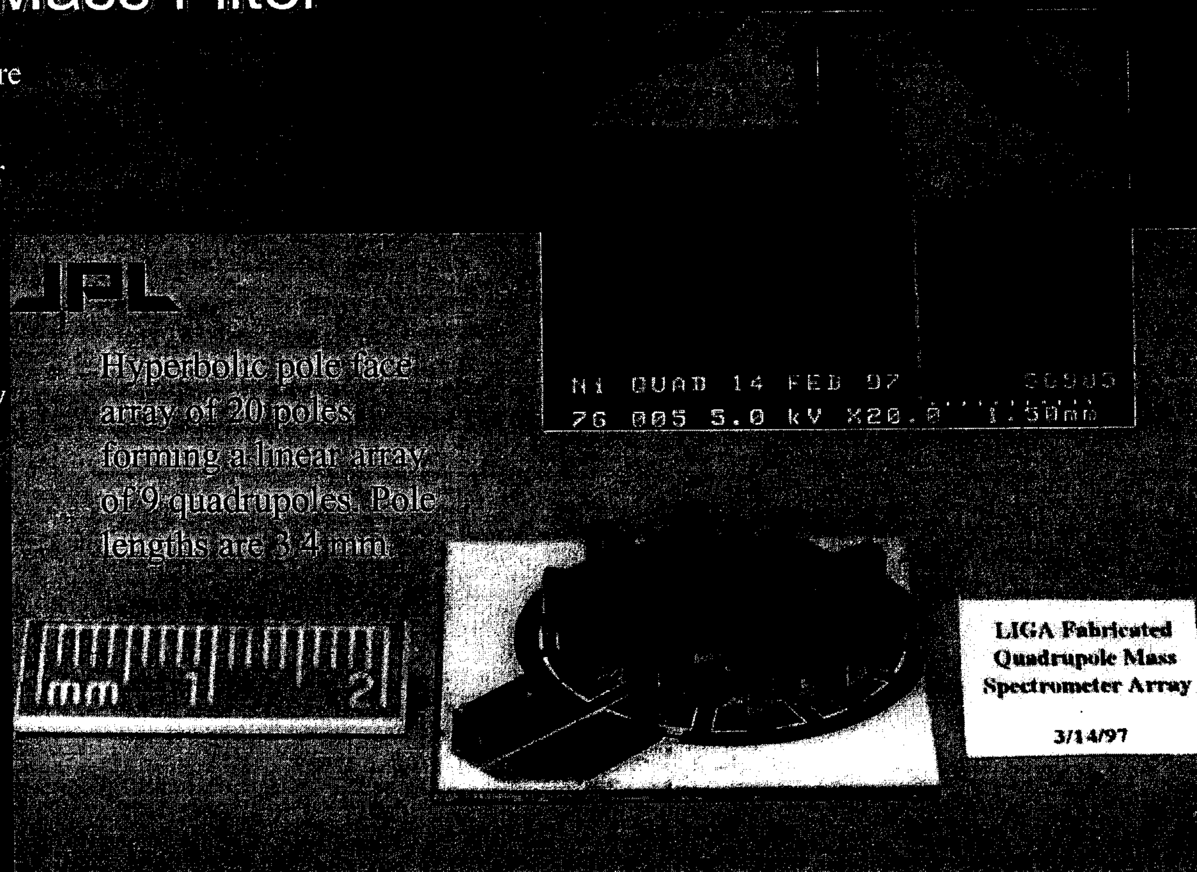

**March 25-28, 2002**

**Pasadena, CA**

# LIGA Fabricated Linear Quadrupole Array

- Quadrupole Mass Filter



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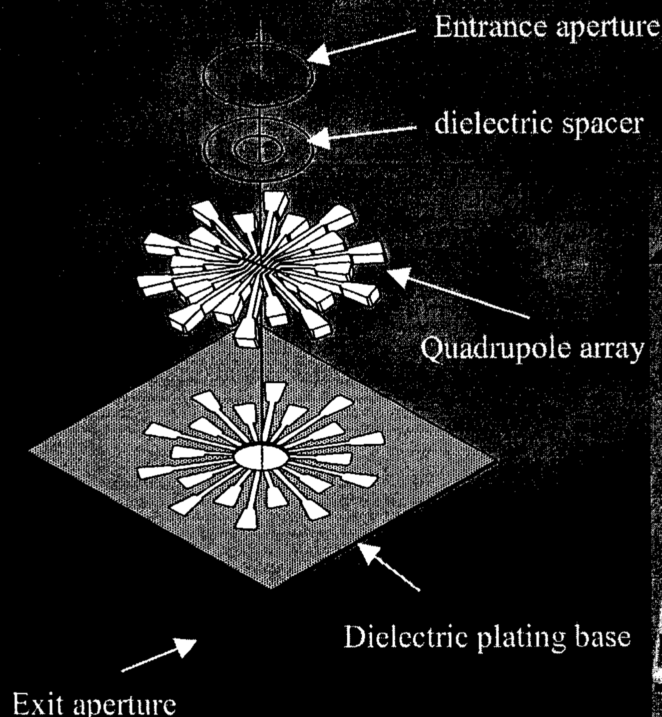


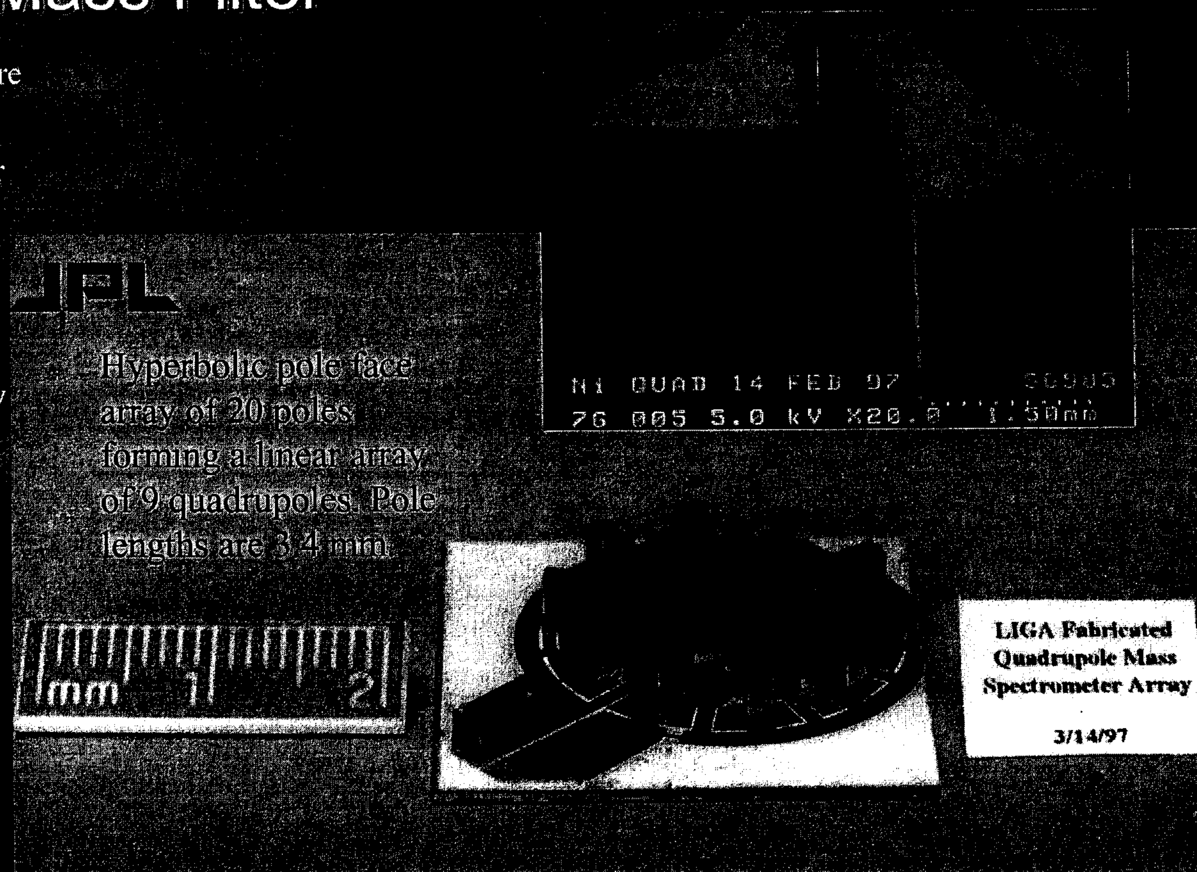

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# The 3rd Workshop on Harsh-Environment Mass Spectrometry

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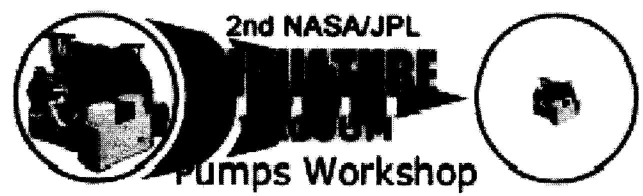


March 25-28, 2002

Pasadena, CA

ELLIE TREVARTHEN / HEMS 2002
JET PROPULSION LABORATORY
4800 OAK GROVE DRIVE, M/S 306-463
PASADENA, CA 91109-8099

**Purpose:** In situ mass spectrometry (MS) in a wide variety of harsh environments—from outer space to Earth's oceans to battlefield scenarios—is rapidly becoming a reality. There are many common features to MS deployment in these vastly different conditions, including high reliability, small size, and low power requirements. The Harsh-Environment Mass Spectrometry (HEMS) Workshop will encourage interaction among those working on deployment of mass spectrometers in various harsh environments. The Miniature Pumps Workshop is inspired by the surge in development of miniaturized vacuum-dependent instrumentation such as mass spectrometers, charged particle analyzers, electron columns, and sublimation cells, to cite a few examples.

**Technical Program:** Talks/posters for the HEMS Technical Sessions will be selected for their focus on making mass spectrometer components and systems rugged and portable; interfacing mass spectrometers to the environment; autonomous sampling strategies; unattended operations; adaptive sampling; data processing and communications; enabling technologies; and miniaturization. The focus for the Miniature Pumps Workshop will be applications requirements, state-of-the-art pumping technology, the fundamental operating characteristics of different approaches, and technological limits to performance with decreasing size and mass. Rough and high-vacuum technologies will be covered, with an emphasis on miniaturization. Each session (except Session IV) will begin with an invited speaker.

**Abstracts:** Those interested in presenting a poster or talk should submit an abstract (maximum 500 words, in English). **Submission deadline: December 15, 2001.** Submission instructions are available on the Workshop website. The Proceedings will be distributed at the workshop. Please contact Ellie Trevarthen if you cannot access the website.

**General Information:** The workshop will be held at the Courtyard Los Angeles Old Pasadena, 180 North Fair Oaks Avenue, Pasadena, CA 91103, Ph: (626) 403-7600, Fax: (626) 403-7700. It is 40 miles from the Los Angeles Int'l. Airport, 12 miles from the Burbank Airport. The Courtyard Old Pasadena is in the heart of the revitalized "Old Town" Pasadena Historic District; a short walk from your room you will find many shopping, dining, and entertainment choices. More information and directions can be found at http://Courtyard.com/.

**Registration:** $150 for two-day HEMS Workshop, $75 for one-day Miniature Pumps Workshop, $225 for both Workshops. Download the registration form from the conference website. **Payment: by credit card or bank draft (in U.S. Dollars, drawn on a U.S. bank).** The fee includes Workshop costs, Proceedings, continental breakfast and lunch each day, and Wednesday evening dinner (transportation and accommodations not included). **Registration deadline: February 1, 2002. Refund for cancellation: request must be received by February 1, 2002** ($50 processing fee will be deducted). Registration is limited to 100 participants.

**Accommodations:** A block of rooms has been reserved at the conference hotel at the rate of **$99 per night**. *Please contact the hotel directly to make your reservation*; mention **"HEMS 2002"** to receive the conference rate. The hotel may fill up quickly, so we encourage you to make your reservation early. For other hotels in the area, contact Ellie Trevarthen.

**Sponsors:** Corporations interested in participating in the vendor exhibit should complete the ***Corporate Sponsor Form*** at the conference website and submit it **to Ellie Trevarthen by December 15, 2001.** Contact Ellie Trevarthen for assistance, if needed.

* * * * * * * * * * * * * * * * * * * * * * * *

### WORKSHOP CONTACTS


Website: http://cot.marine.usf.edu/hems/HEMSconf.htm

Technical Program: Tim Short <tshort@seas.marine.usf.edu>, Dean.Wiberg@jpl.nasa.gov, Patricia.Beauchamp@jpl.nasa.gov, Jack Beauchamp <jlbchamp@cco.caltech.edu>

Registration: conf.admin@jpl.nasa.gov

Abstract Submission / Logistics: Ellie.Trevarthen@jpl.nasa.gov


## TENTATIVE AGENDA

### Monday, March 25 — TRAVEL DAY

7:00 P.M. WELCOME RECEPTION AT COURTYARD OLD PASADENA

### Tuesday, March 26 — HEMS WORKSHOP

8:00 A.M. CONTINENTAL BREAKFAST

8:30 A.M. TECHNICAL SESSION I: SPACE ENVIRONMENTS
**INVITED SPEAKER:** JACK BEAUCHAMP, CALIFORNIA INSTITUTE OF TECHNOLOGY
**TOPIC:** "NOVEL MASS SPECTROMETRIC APPROACHES TO THE IN SITU CHEMICAL ANALYSIS OF GALACTIC AND COMETARY DUST PARTICLES"

12:00 NOON INFORMAL LUNCH BUFFET

1:30 P.M. TECHNICAL SESSION II: MASS SPECTROMETERS FOR UNDERWATER APPLICATIONS
**INVITED SPEAKER:** JOHN DELANEY, UNIVERSITY OF WASHINGTON
**TOPIC:** "THE NEPTUNE PROJECT: AN INTERACTIVE EARTH-OCEAN OBSERVATORY AT THE SCALE OF A TECTONIC PLATE"

4:30 P.M. POSTER SESSION

6:00 P.M. EVENING FREE

### Wednesday, March 27 — HEMS WORKSHOP

8:00 A.M. CONTINENTAL BREAKFAST

9:30 A.M. TECHNICAL SESSION III: EARTH ENVIRONMENTS
**INVITED SPEAKER:** HENK MEUZELAAR, UNIVERSITY OF UTAH
**TOPIC:** "MAPPING AND MONITORING COMPLEX CHEMICAL COMPONENTS IN AMBIENT AIR USING FAST GC/MS AND MULTIVARIATE DATA ANALYSIS"

12:00 NOON INFORMAL LUNCH BUFFET / VENDOR EXPO

1:30 P.M. TECHNICAL SESSION IV: BIO-APPLICATIONS

3:00 P.M. TECHNICAL SESSION V: NOVEL CONCEPTS / MINIATURIZATION
**INVITED SPEAKER:** ARA CHUTJIAN, JET PROPULSION LABORATORY
**TOPIC:** "MINIATURE MASS SPECTROMETERS AND FRONT-END INTERFACES"

7:00 P.M. RECEPTION/CONFERENCE DINNER, COURTYARD OLD PASADENA
GUEST SPEAKER: DR. CHARLES ELACHI, DIRECTOR, JET PROPULSION LABORATORY, "SPACE EXPLORATION IN THE NEXT DECADE"

### Thursday, March 28 — PUMPS WORKSHOP

8:00 A.M. CONTINENTAL BREAKFAST

8:30 A.M. TECHNICAL SESSION I: MINIATURIZATION / TECHNICAL ISSUES
**INVITED SPEAKER:** PHIL MUNTZ, UNIVERSITY OF SOUTHERN CALIFORNIA
**TOPIC:** "THE TECHNICAL ISSUES ASSOCIATED WITH HIGHLY MINIATURIZED VACUUM SYSTEMS"

12:00 NOON INFORMAL LUNCH BUFFET

1:30 P.M. TECHNICAL SESSION II: COMMERCIALIZATION ISSUES
**INVITED SPEAKER:** PETER KARDOK, ALCATEL
**TOPIC:** "THE ISSUES LIMITING LARGE-SCALE COMMERCIALIZATION OF MINIATURE VACUUM SYSTEMS"

5:00 P.M. CLOSING REMARKS

5:30 P.M. ADJOURN